U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

PB-253 941

SAMPLING AND ANALYSIS OF SELECTED TOXIC SUBSTANCES

TASK IB. HEXACHLOROBUTADIENE

MIDWEST RESEARCH INSTITUTE

PREPARED FOR
ENVIRONMENTAL PROTECTION AGENCY

JUNE 1976

176031

EPA 560/6-76-015

PB 253 941

SAMPLING AND ANALYSIS OF SELECTED TOXIC SUBSTANCES

Task IB - Hexachlorobutadiene

Environmental Protection Agency
Office of Toxic Substances
Washington, D.C. 20460

June, 1976

REPRODUCED BY
NATIONAL TECHNICAL
INFORMATION SERVICE
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA

(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA 560/6-76-015	2.	3. RECIPIENT'S ACCESSION NO. PB253941
4. TITLE AND SUBTITLE Sampling and Analysis of Selected Toxic Substances Task IB - Hexachlorobutadiene		5. REPORT DATE June 1976
		6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) R. T. Li, J. E. Going, and J. L. Spigarelli		8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Midwest Research Institute 425 Volker Boulevard Kansas City, Missouri 64110		10. PROGRAM ELEMENT NO.
		11. CONTRACT/GRANT NO. Contract No. 68-01-2646
12. SPONSORING AGENCY NAME AND ADDRESS Environmental Protection Agency Office of Toxic Substances Washington, D.C. 20460		13. TYPE OF REPORT AND PERIOD COVERED Final, July 1974-September 1975
		14. SPONSORING AGENCY CODE

15. SUPPLEMENTARY NOTES

16. ABSTRACT

A sampling and analysis program was conducted to determine the extent of environmental contamination by hexachlorobutadiene around nine chemical manufacturers. The plants selected represented six major industries: perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, triazine herbicides, and pentachloronitrobenzene. Air, water, soil, and sediment samples were collected on and around the plants.

In general, higher levels of HCBD in air were associated with production of perchloroethylene and trichloroethylene while the level in the vicinity of chlorine and triazine herbicide plants was very low. No HCBD was found in the air at the pentachloronitrobenzene plant. The highest level of HCBD in air and soil was at a plant using on-site landfill and open pit storage waste-disposal methods. High levels were detected in loading and transfer areas at plants using off-site disposal methods.

The highest level of HCBD found in the air on plant property was 463 $\mu g/m^3$. Levels of HCBD in open wastewater treatment streams were as high as 244 and 75 μg/liter at two plants. Soil levels of HCBD reached 980 and 29 $\mu g/g$ at two plants.

(continued)

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
Monitoring - Air, Water, Soil, Sediment	Hexachlorobutadiene	Organic Chemistry

PRICES SUBJECT TO CHANGE

18. DISTRIBUTION STATEMENT Releast Unlimited	19. SECURITY CLASS *(This Report)* Unclassified	21. NO. OF PAGES 152
	20. SECURITY CLASS *(This page)* Unclassified	22. PRICE 6.75-2.25

EPA Form 2220-1 (9-73)

EPA-560/6-76-015

SAMPLING AND ANALYSIS OF SELECTED TOXIC SUBSTANCES

Task IB - Hexachlorobutadiene

Contract No. 68-01-2646

Project Officer

William A. Coniglio
Office of Toxic Substances
Environmental Protection Agency
Washington, D.C. 20460

Prepared for

Environmental Protection Agency
Office of Toxic Substances
Washington, D.C. 20460

June 1976

ib

NOTICE

This report has been reviewed by the Office of Toxic Substances, Environmental Protection Agency, and approved for publication. Approval does not signify that the contents necessarily reflect the views and policies of the Environmental Protection Agency. Mention of trade names or commercial products is for purposes of clarity only and does not constitute endorsement or recommendation for use.

ic

CONTENTS

Page

Summary 1

Section

I Introduction 3

II Experimental Procedures 5

Sampling Procedures 5
Analysis Procedures 5

III Selection of Sampling Sites 8

Selection Criteria 8
Recommended Sampling Sites 10

IV Presampling Surveys and Field Sampling 14

Presampling Surveys 14
Field Sampling 14

V Discussion of Results and Conclusions 19

Vulcan Materials Company, Wichita, Kansas 19
Linden Chlorine Plant, Linden, New Jersey 28
Stauffer Chemical Company, Louisville, Kentucky . . . 30
Dow Chemical Company, Pittsburg, California 37
E. I. du Pont de Nemours and Company, Inc.,
Corpus Christi, Texas 41
Diamond Shamrock Corporation, Deer Park, Texas . . . 44
Ciba-Geigy Corporation, St. Gabriel, Louisiana . . . 49
Olin Corporation, McIntosh, Alabama 51
PPG Industries, Inc., Lake Charles, Louisiana 53

CONTENTS (concluded)

Section Page

VI Summary and Conclusions for Program Task IB 60

Summary 60
Conclusions 63

References 64

Appendix A - Presampling Survey and Field Sampling 65

Appendix B - Analytical Data 118

Appendix C - Method Development for Sampling and Analysis 131

FIGURES

No.		Page
1	Air Sampling Train	6
2	Geographic Location of Recommended Sampling Sites	12
3	Presampling Survey and Field Sampling Schedule	15
4	Average Concentrations of HCBD in Air Per 20-Hr Period at 18 Sampling Stations at Vulcan Materials Company, Wichita, Kansas	21
5	HCBD Concentration Per 20-Hr Period at Sampling Stations Within the Plant Perimeter (Vulcan)	22
6	HCBD Concentration Per 20-Hr Period, Downwind Stations (Vulcan)	23
7	HCBD Concentration Per 20-Hr Period, Upwind Stations (Vulcan)	24
8	Average Concentrations of HCBD in Air at Nine Sampling Stations at Stauffer Chemical Company, Louisville, Kentucky	33
9	HCBD Concentration Per 24-Hr Period at Sampling Stations Around the Plant (Stauffer)	34
10	Average Concentrations of HCBD in Air at Eight Sampling Stations at Dow Chemical Company, Pittsburg, California	38
11	Average Concentrations of HCBD in Air at Five Sampling Stations at E. I. du Pont de Nemours Company, Corpus Christi, Texas	42
12	Average Concentrations of HCBD in Air at Eight Sampling Stations at Diamond Shamrock Corporation, Deer Park, Texas	47

FIGURES (concluded)

No.		Page
13	Average Concentrations of HCBD in Air at Eight Sampling Stations at Ciba-Geigy Corporation, St. Gabriel, Louisiana	50
14	Average Concentrations of HCBD in Air at Eight Sampling Stations.	52
15	Average Concentrations of HCBD in Air at 10 Sampling Stations at PPG Industries, Lake Charles, Louisiana . . .	55
16	Summary of HCBD Concentrations in Air	62
A-1	Presampling Survey and Field Sampling Schedule.	67
A-2	Sampling Locations at Vulcan Materials Company - Wichita Plant	69
A-3	Vulcan Materials Company - Wichita Plant, and Surrounding Areas	72
A-4	Sampling Locations at Stauffer Chemical Company - Louisville, Kentucky.	78
A-5	Sample Locations at Dow Chemical Company, Pittsburg, California.	85
A-6	Sample Locations at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas	90
A-7	Sampling Locations at Diamond Shamrock Corporation, Deer Park, Texas.	97
A-8	Sample Locations at Ciba-Geigy Corporation, St. Gabriel, Louisiana	101
A-9	Sampling Locations at Olin Corporation, McIntosh, Alabama .	107
A-10	Sampling Locations at PPG Industries, Lake Charles, Louisiana	114
C-1	Apparatus for Recovery of HCBD From Water by Vaporization at Reduced Pressure	135

TABLES

No.		Page
1	Estimated Quantities of HCBD Present in Industrial Wastes, By-Products, And Products in 1972	9
2	Production Capacity, Process Technology and Waste Disposal at Recommended Sites	13
3	Field Sampling Summary	17
4	Summary of Air Sampling Parameters	18
5	Comparison of HCBD Levels at 4 and 11 ft for Five Time Periods	25
6	HCBD Concentrations in Soil and Sediment From Vulcan Materials Company, Wichita, Kansas	27
7	HCBD Concentrations in Water From Vulcan Materials Company, Wichita, Kansas	29
8	HCBD Concentrations in Water and Solids From Linden Chlorine Company, Linden, New Jersey	31
9	HCBD Concentrations in Soil and Sediment From Stauffer Chemical Company, Louisville, Kentucky	36
10	HCBD Concentration in Water From Stauffer Chemical Company, Louisville, Kentucky	36
11	HCBD Concentrations in Soil From Dow Chemical Company, Pittsburg, California	40
12	HCBD Concentrations in Soil and Sediment From E. I. du Pont de Nemours and Company, Corpus Christi, Texas	43
13	HCBD Concentrations in Water From E. I. du Pont de Nemours and Company, Corpus Christi, Texas	45

TABLES (continued)

No. Page

14 HCBD Concentrations in Soil From Diamond Shamrock Corporation, Deer Park, Texas 48

15 HCBD Concentrations in Water From Diamond Shamrock Corporation, Deer Park, Texas 48

16 HCBD Concentrations in Soil and Sediment, Olin Corporation, McIntosh, Alabama 54

17 HCBD Concentrations in Soil and Sediment From PPG Industries, Lake Charles, Louisiana. 57

18 HCBD Concentrations in Water From PPG Industries, Lake Charles, Louisiana. 58

19 Data Summary for Program Task No. 1 61

A-1 Air Sampling Data at Vulcan Materials Company, Wichita, Kansas. 70

A-2 Plant Activities During Sampling at Vulcan Materials Company, Wichita, Kansas. 73

A-3 Weather Conditions During Sampling at Vulcan Materials Company, Wichita, Kansas. 74

A-4 Air Sampling Data at Stauffer Chemical Company, Louisville, Kentucky. 79

A-5 Plant Activities During Sampling at Stauffer Chemical Company, Louisville, Kentucky 81

A-6 Weather Conditions During Sampling at Stauffer Chemical Company, Louisville, Kentucky 82

A-7 Air Sampling Data at Dow Chemical Company, Pittsburg, California. 86

A-8 Weather Conditions and Plant Activities During Sampling at Dow Chemical Company, Pittsburg, California. 87

TABLES (continued)

No. Page

A-9 Air Sampling Data at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas 91

A-10 Plant Activities During Sampling at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas 93

A-11 Weather Conditions During Sampling at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas 94

A-12 Air Sampling Data at Diamond Shamrock Corporation, Deer Park, Texas 98

A-13 Weather Conditions During Sampling at Diamond Shamrock Corporation, Deer Park, Texas. 99

A-14 Air Sampling at Ciba-Geigy Corporation, St. Gabriel, Louisiana. 102

A-15 Weather Conditions During Sampling at Ciba-Geigy, St. Gabriel, Louisiana 104

A-16 Air Sampling Data at Olin Corporation, McIntosh, Alabama . 108

A-17 Weather Conditions During Sampling at Olin Corporation, McIntosh, Alabama. 110

A-18 Air Sampling Data at PPG Industries, Lake Charles, Louisiana. 115

A-19 Weather Conditions During Sampling at PPG Industries, Lake Charles, Louisiana. 117

B-1 HCBD Concentrations in Air Samples From Vulcan Materials Company, Wichita, Kansas 119

B-2 HCBD Concentrations in Air Samples From Stauffer Chemical Company, Louisville, Kentucky. 124

B-3 HCBD Concentrations in Air Samples From Dow Chemical Company, Pittsburg, California 126

B-4 HCBD Concentrations in Air Samples From E. I. du Pont de Nemours and Company, Corpus Christi, Texas 127

TABLES (concluded)

No. Page

B-5 HCBD Concentrations in Air Samples From Diamond Shamrock Corporation, Deer Park, Texas 128

B-6 HCBD Concentrations in Air Samples From Ciba-Geigy Corporation, St. Gabriel, Louisiana 129

B-7 HCBD Concentrations in Air Samples From PPG Industries, Lake Charles, Louisiana 130

C-1 Recovery Studies of HCBD by n-Hexane Extraction 133

C-2 Recovery of HCBD From Water by Concentration on XAD-4 . . . 133

C-3 Loss of HCBD Due to Vaporization. 134

C-4 Recovery of HCBD From Water-Saturated Air 136

C-5 Recovery of HCBD From Sediments 137

The maximum air concentration of HCBD off plant property was 22 $\mu g/m^3$. A level of 10 $\mu g/m^3$ was detected at the boundary of another plant. The highest levels in water off plant property were 23 and 10 μg/liter. A level of 0.11 $\mu g/g$ HCBD in soil was found off plant property and levels of 0.15 and 0.34 $\mu g/g$ were found at the boundary of two other plants.

SUMMARY

The purpose of this program is to provide sampling and analysis capabilities to EPA's Office of Toxic Substances, so that the levels of suspected toxic substances in air, water, soil, and sediment at designated locations throughout the United States may be determined. Four tasks have been assigned on this program. The first task was the sampling and analysis for hexachlorobutadiene (HCBD).

Methods for sampling and analyzing HCBD in air, water, soil, and sediments were evaluated. A protocol was developed and approved.

Nine industrial plants were selected for sampling. The plants represent six major industries: perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, triazine herbicides, and pentachloronitrobenzene.

In general, of the six industries sampled, higher concentrations of HCBD were associated with the production of perchloroethylene, trichloroethylene, and carbon tetrachloride. In the one plant that produced only carbon tetrachloride, however, the HCBD levels were quite low. No HCBD was detected in samples from the pentachloronitrobenzene production plant. The levels of HCBD associated with plants producing chlorine and triazine herbicides were very low.

Several different waste-disposal methods were used at the perchloro- and trichloroethylene plants that were sampled. The highest level of HCBD was detected in air and soil at the plant using on-site landfill and open pit storage. High HCBD levels were detected in loading and transfer areas at plants using off-site disposal methods. Lower levels of HCBD were found at plants using on-site incineration but downwind air concentrations were still elevated above background at both plants. The production of perchloro- and trichloroethylene by low temperature oxychlorination and the incineration of liquid bottom wastes resulted in a low HCBD level in the air but high levels in the effluent water. Levels of 10 μg/liter HCBD were found in treated process water more than 3,000 ft from the plant effluent.

The highest level of HCBD found in the air on plant property was 463 μg/m^3. Levels of HCBD in open wastewater treatment streams were as high as 244 and 75 μg/liter at two plants. Soil levels of HCBD reached 980 and 29 μg/g at two plants.

The maximum air concentration of HCBD off plant property was 22 μg/m^3. A level of 10 μg/m^3 was detected at the boundary of another plant. The highest levels in water off plant property were 23 and 10 μg/liter. A level of 0.11 μg/g HCBD in soil was found off plant property and levels of 0.15 and 0.34 μg/g were found at the boundary of two other plants.

SECTION I

INTRODUCTION

Environmental contamination of HCBD has been reported in the United States. HCBD has been found in fish samples taken from the lower Mississippi River.[1] In addition, HCBD has been detected in waste streams and solid waste disposal sites near chemical manufacturing sites.[1]

On July 5, 1973, Midwest Research Institute (MRI) initiated a study to estimate the quantities and identify sources of HCBD in the environment. The origin of HCBD in the environment in the United States was identified as the waste materials or by-products from the production of perchloroethylene, trichloroethylene, carbon tetrachloride, chlorine, pentachloronitrobenzene and the herbicide Dacthal®. Specific industrial plants from the above industries were recommended to EPA as potential sampling sites.

On June 27, 1974, the current MRI project (3953-C) entitled "Sampling and Analysis of Selected Toxic Substances" was initiated. The objective of this program is to provide the EPA with sampling and analysis capabilities to determine the levels of toxic substances in air, water, soil and sediment from designated sources and ambient locations throughout the United States. The first task of this program was the sampling and analysis for HCB and HCBD. Tasks II, III, and IV of this program are the sampling and analysis for ethylene dibromide, evaluation of vinyl chloride levels in outdoor and indoor air due to the presence of PVC products and additional sampling and analysis for ethylene dibromide, respectively. The Task II ethylene dibromide study has been completed and reported to the Office of Toxic Substances in September 1975 under the title of: "Sampling and Analysis of Selected Toxic Substances: Task II - Ethylene Dibromide," EPA Report No. 560/6-75-001. The Task III study has been completed and reported to the Office of Toxic Substances in April 1975 under the title "Sampling and Analysis of Selected Toxic Substances: Task III - Vinyl Chloride, Secondary Sources," EPA Report No. 560/6-76-002.

This report describes Task IA of the program, i.e., the sampling and analysis for HCBD as follows: experimental procedures; screening and selection of sampling sites; presampling surveys and field sampling; and discussion of results and conclusions. Site surveys and field sampling data for individual sites, analytical data, and methods development efforts are appended to the report.

SECTION II

EXPERIMENTAL PROCEDURES

SAMPLING PROCEDURES

Water was sampled by two different methods--grab sampling and porous polymer extraction. The grab water samples were composited and stored in glass 1-gal. bottles that had been used for pesticide grade solvents. In the second technique, water was mechanically pumped through a 30 x 5.2 cm i.d. glass tube packed with 250 g Amberlite XAD-4. The Amberlite resin removed HCBD quantitatively from the water stream flowing at 0.6 to 1.4 liters/min. The composited water samples and the Amberlite resin tubes were capped and stored in ice chests until ready for analysis.

Air was sampled through a 37 mm diameter, 0.8 μm pore size, millipore filter, followed by a 15 cm, 1.2 cm i.d., glass sampling tube* packed with 1 g Tenax®-GC. Air was drawn through the filter and Tenax®-GC tube with the aid of a mechanical pump. The flow rate was regulated with either an 18 gauge needle (~ 3.5 liters/min) or a 26 gauge needle (~ 0.4 liters/min). A schematic of the air sampling train is shown in Figure 1.

Soil and sediment samples from the top 2 to 5 cm were collected at designated sites. From 0.5 to 1 kg of sample was composited and stored in wide-mouth glass bottles with Teflon®-lined caps and kept in an ice chest until ready for analysis.

ANALYSIS PROCEDURES

Sample Preparation

The millipore filter and the Tenax®-GC resin for each air sample was sequentially extracted with 20, 20, and 10 ml of pesticide grade hexane using an ultrasonic water bath. During the extraction, ice was added to the ultrasonic bath to minimize evaporative loss of HCBD. The hexane extracts were combined and diluted to 50.0 ml.

---

* In sampling some industrial plants, two Tenax®-GC tubes were used in tandem.

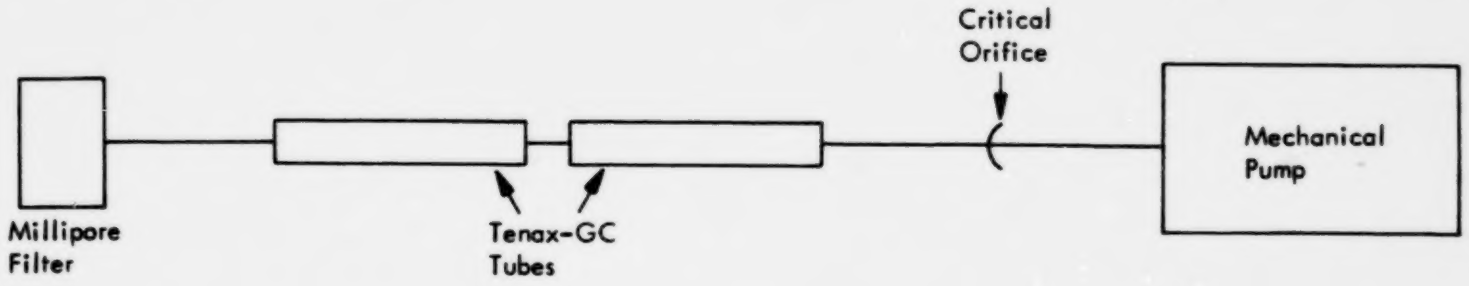

Critical
Orifice
Millipore
Filter
Tenax-GC
Tubes
Mechanical
Pump

Figure 1. Air sampling train

The soil samples were first sifted on a U.S. Standard No. 18 sieve to remove stones and other foreign material. A 100 g sample was then extracted with 100 ml of n-hexane in a soxhlet apparatus overnight. The hexane extracts were transferred to 100 ml volume flasks and diluted to volume. A similar sample preparation procedure was used on the sediment samples except that the sifting step was omitted.

A 500 to 1,000 ml portion of each grab water sample was extracted sequentially with 20, 20, and 10 ml of hexane. The extracts were collected in a 50 ml volumetric flask and diluted to volume. The Amberlite XAD-4 resin was extracted with 250 ml hexane using a soxhlet apparatus. The extract was collected in a 250 ml volumetric flask and diluted to volume.

All of the extracted samples were kept in a walk-in cold room maintained at 4°C. Prior to analysis, the samples were brought to room temperature and diluted or concentrated as necessary for analysis.

Instrumentation and Conditions

A Microtek-2000R gas chromatograph equipped with an electron capture (tritium) detector was used. The output of the gas chromatograph was connected to a Hewlett-Packard 3380A integrator-recorder, which provides a printout of the chromatogram with integrated areas of individual peaks and respective retention times. A 4 ft x 1/4 in. glass column packed with 1.5% OV-17/1.95% QF-1 coated on 100/120 mesh Supelcoport® was used for analysis. The chromatographic operating conditions were: injector temperature, 200°C; column temperature, 100°C; detector temperature, 180°C; carrier flow rate, 50 ml/min nitrogen; purge flow rate, 90 ml/min nitrogen; and detector voltage, 10 V DC.

The instrumental limit of detection for HCBD at the above mentioned conditions was 1 pg (10^{-12} g). Therefore, as an example, for any amount of air sampled, the quantity of HCBD in the sample required for detection was greater than 5 ng (based on 10 µl injections of a 50 ml solution).

Calibration

A 10 ng/ml composite standard solution of HCBD was used to obtain the calibration curves. The standard solution was prepared by dilution of a stock solution made up from EPA reference standards obtained from Pesticides and Toxic Substances Effects Laboratory, National Environmental Research Center, Research Triangle Park, North Carolina. Concentration ranges chosen for the calibration curve were from 10 to 60 pg, and linearity was observed.

A new calibration curve was obtained daily for the sample analysis. During the day, a known amount of the standard was injected periodically into the GC to check for changes in retention time and peak intensity.

SECTION III

SELECTION OF SAMPLING SITES

The objective of this task was to determine environmental levels of HCBD by the sampling and analysis of samples from selected industrial plants. Therefore, it was important that the selected sampling locations be representative of the total industrial locations that are sources of HCBD.

SELECTION CRITERIA

Selection criteria were chosen to achieve representative sampling of sites that are most likely to have detectable quantities of HCBD present.

The criteria used for the selection of industrial plants for sampling of HCBD are:

* Estimated quantity of HCBD in industrial wastes, products, and by-products.

* Methods of production.

* Methods of waste disposal.

* Geographic location of the industrial plants.

Estimated Quantity of HCBD in Industrial Wastes, Products, and By-Products

In 1974, there was no manufacturer of HCBD in this country. However, industry sources report that HCBD contained in the "heavy ends" waste materials (residues) in the production of many chlorinated organic compounds, as well as in the electrolytic processes (either diaphragm or mercury cells) for chlorine gas when graphite anodes are used. An estimation of the amount HCBD produced in industrial wastes, by-products, and products is given in Table 1. As indicated in Table 1, over 99% of the HCBD contamination in the environment was estimated to be from the perchloroethylene, trichloroethylene, and carbon tetrachloride industries.2/

Table 1. ESTIMATED QUANTITIES OF HCBD PRESENT IN INDUSTRIAL WASTES, BY-PRODUCTS, AND PRODUCTS IN 1972[2]

Product	U.S. Production in 1972 (tons)	HCBD (tons)
Perchloroethylene	367,400	3,251
Trichloroethylene	213,500	1,132
Carbon tetrachloride	498,500	1,047
Chlorine	9,538,000	26
Dacthal®	1,000	0
Vinyl chloride	2,545,000	0
Atrazine, propazine, simazine	56,000	0
Pentachloronitrobenzene	1,500	0
Mirex	500	0

Therefore, industrial plants that produce these chemicals were given high priority in the selection of sampling sites.

Method of Production

The production method affects the quantity of HCBD formed as a by-product. Therefore, the potential environmental contamination is dependent upon the production method. For example, carbon tetrachloride, perchloroethylene and trichloroethylene are produced in several ways. If chlorine and the respective aliphatic hydrocarbons are fed into a high-temperature reactor and the products are collected by distillation, HCBD is discharged as a by-product in the "heavy ends" wastes. However, if the production of carbon tetrachloride involves the reaction of chlorine with carbon disulfide, coproducts or by-products other than reusable sulfur are greatly reduced.

Methods of Waste Disposal

Disposal methods for "heavy ends" wastes played a role in the selection of plants for sampling. The selected plants used a variety of disposal methods including landfill, deep well, sealed lagoons, on-site incineration, and shipment of wastes to other disposal firms.

Geographic Location of the Industrial Plant

Industrial plants were selected from across the country to determine whether the potential for environmental contamination was a national problem.

RECOMMENDED SAMPLING SITES

Using these general criteria as a guide, 10 industrial plants were selected for inclusion in this study. These industrial plants are listed below.

Perchloroethylene

Stauffer Chemical Company	Louisville, Kentucky
Vulcan Materials Company	Wichita, Kansas

Trichloroethylene

PPG Industry, Inc.	Lake Charles, Louisiana
Diamond Shamrock Corporation	Deer Park, Texas

Carbon tetrachloride

E. I. du Pont de Nemours and Company, Inc.	Corpus Christi, Texas
Dow Chemical Company	Pittsburg, California

Chlorine

Linden Chlorine	Linden, New Jersey
Kaiser Aluminum and Chemical Corporation	Gramercy, Louisiana

Triazine herbicides (atrazine, propazine, simazine)

Ciba-Geigy Corporation	St. Gabriel, Louisiana

Pentachloronitrobenzene

Olin Corporation	McIntosh, Alabama

The geographic location and EPA region of these plants are shown in Figure 2.

During the process of selecting the sampling sites, efforts were made to select industrial plants that produce a unique product rather than a combination of several products. The efforts were successful for the two chlorine plants selected. However, plants producing low molecular weight chlorinated hydrocarbons do not generally produce a single product. All five plants that produce trichloroethylene also produce perchloroethylene. Fortunately, not all the perchloroethylene production plants produce trichloroethylene. However, these plants also produce carbon tetrachloride. The annual production capacity (1972), process technology, and latest waste disposal methods for each of the sampling sites are presented in Table 2.

The Dacthal® production facility (Diamond Shamrock Corporation) in Greens Bayou, Texas, was not selected for sampling because the waste handling and product contamination were substantially changed from pre-1972 procedures.

Vinyl chloride and mirex production facilities were omitted from this survey.

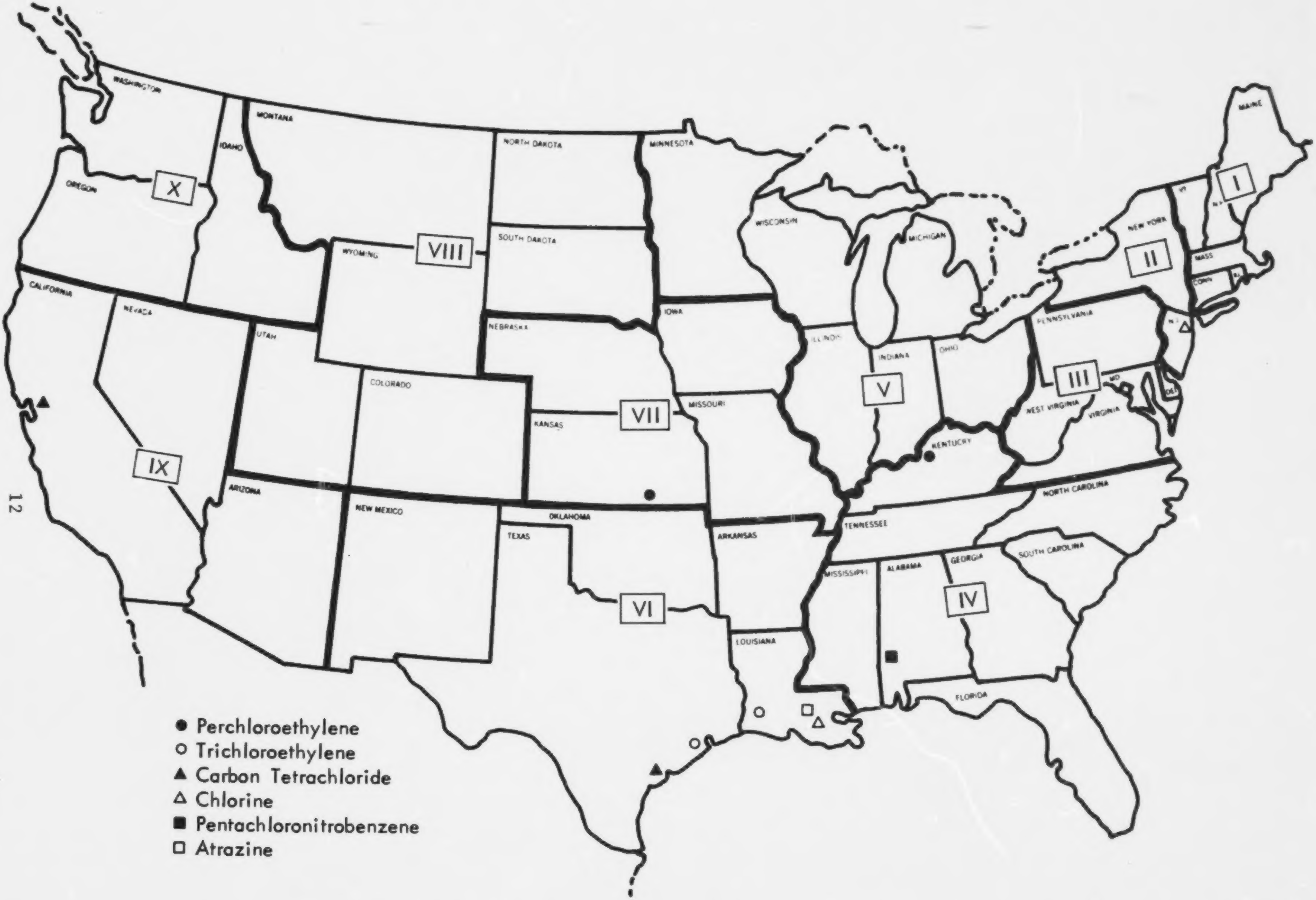

I
II
III
IV
V
VI
VII
VIII
IX
X
WASHINGTON
OREGON
CALIFORNIA
NEVADA
IDAHO
MONTANA
WYOMING
UTAH
ARIZONA
COLORADO
NEW MEXICO
NORTH DAKOTA
SOUTH DAKOTA
NEBRASKA
KANSAS
OKLAHOMA
TEXAS
MINNESOTA
IOWA
MISSOURI
ARKANSAS
LOUISIANA
WISCONSIN
ILLINOIS
MICHIGAN
INDIANA
OHIO
KENTUCKY
TENNESSEE
MISSISSIPPI
ALABAMA
GEORGIA
FLORIDA
SOUTH CAROLINA
NORTH CAROLINA
VIRGINIA
WEST VIRGINIA
MD
DEL
PENNSYLVANIA
N.J.
NEW YORK
VT
N.H.
MAINE
MASS
CONN
R.I.
● Perchloroethylene
○ Trichloroethylene
▲ Carbon Tetrachloride
△ Chlorine
■ Pentachloronitrobenzene
□ Atrazine

Figure 2. Geographic location of recommended sampling sites

Table 2. PRODUCTION CAPACITY, PROCESS TECHNOLOGY AND WASTE DISPOSAL AT RECOMMENDED SITES

Producers	Production sites	EPA region	Annual production capacity (10^3 tons)	Waste disposal	Process technology[a]
Perchloroethylene					
Stauffer Chemical Company	Louisville, Kentucky	IV	35	HCB recovered for sale, remainder recycled to chlorinator	Chlorination with low molecular weight hydrocarbons, e.g., ethane, propane
Vulcan Materials Company	Wichita, Kansas	VII	25	Earth-covered groundfill	
Trichloroethylene					
PPG Industry Company	Lake Charles, Louisiana	VI	140	Incineration, landfill	Ethylene and chlorine as raw materials, under catalytic reaction at 250 to 300°C
Diamond Shamrock Corporation	Deer Park, Texas	VI	60	Ship to Rollins International for incineration	
Carbon tetrachloride					
E. I. du Pont de Nemours and Company, Inc.	Corpus Christi, Texas	VI	250	Landfill, ship to outside firm for disposal	Chlorination with methane at elevated temperature
Dow Chemical Company	Pittsburg, California	IX	23	Incineration	
Chlorine					
Linden Chlorine Company	Linden, New Jersey	II	66	Discharge to holding pond	Mercury cell; graphite electrode
Kaiser Aluminum and Chemical Corporation	Gramercy, Louisiana	VI	58	Landfill	Diaphragm cell; graphite electrode
Triazine herbicides					
Ciba-Geigy Corporation	St. Gabriel, Louisiana	VI	> 75	Still bottoms incinerated by an outside processor to extinction	Reaction of cyanuric chloride with appropriate amino hydrocarbons at elevated temperature
Pentachloronitrobenzene					
Olin Corporation	McIntosh, Alabama	IV	1.5	Stored in "blocks" covered with plastic sheet	Nitration of pentachlorobenzene or chlorination of various chloronitrobenzenes in the presence of catalyst

---

[a] Kirk-Othmer, Encyclopedia of Chemical Technology 2nd ed., Interscience Publishers, New York, New York (1972).

SECTION IV

PRESAMPLING SURVEYS AND FIELD SAMPLING

To plan the strategy for successful field sampling at the selected industrial plants, a presampling survey was conducted at each plant generally from 2 to 4 weeks prior to sampling. Each presampling survey was arranged through telephone contact with the appropriate plant officials whose names were provided by the EPA project officer. Figure 3 shows the complete schedule for presampling surveys and field sampling.

PRESAMPLING SURVEYS

During the presampling site survey, a plant map was obtained. Information regarding the possible sources of HCBD contamination, the production technology, and waste disposal techniques were obtained. In addition, the production and waste disposal sites, as well as the transportation routes were delineated. Accessible electrical outlets inside the plant were also located for possible use in air sampling. Meterological conditions, such as wind direction and rainfall were investigated. Tentative sampling dates were agreed upon, subject to final confirmation by plant officials prior to the departure of the sampling crew from MRI.

FIELD SAMPLING

Upon the completion of a presampling site survey, the sampling strategy was planned. In general, air sampling stations were positioned upwind and at several distances downwind from the suspected source(s) of contamination. The air samplers were usually positioned 4 ft above ground. When the wind direction was uncertain, stations were positioned around the entire plant area.

Water sampling was conducted at suspected sources of contamination such as upstream and downstream of waste effluent. Storm runoff was collected whenever possible. Water samples from equilization ponds or solar ponds were collected to determine if the ponds were sources of air contamination through liquid vapor equilibrium HCBD.

	May	June	July	August	September
Vulcan Materials Wichita, Ks.	▲ ▬				
Linden Chlorine Linden, N.J.	▲ ▬				
Stauffer Chemical Louisville, Ky.	▲	▬			
Dow Chemical Pittsburg, Calif.		▲		▬	
du Pont Corpus Christi, Tex.			▲	▬	
Diamond Shamrock Deer Park, Tex.			▲	▬	
Ciba-Geigy Corp. St. Gabriel, La.			▲	▬	
Olin Corp. McIntosh, Ala.			▲	▬	
Kaiser Aluminum Gramercy, La.				▲	
PPG Industries Lake Charles, La.				▲	▬

▲ Presampling Visit

▬ Field Sampling

Figure 3. Presampling survey and field sampling schedule

Soil sampling was generally conducted along the plant boundaries, transportation routes, and around waste disposal and storage areas. Sediment samples were collected from streams, equilization ponds and natural solar ponds. Solids and liquid were also sampled from open disposal pits. The preparation for sampling usually was conducted 3 to 5 days prior to the sampling date. Sampling equipment was sent to the plant scheduled for sampling. Major sampling equipment included vacuum pumps, poles, rubber hoses, electrical prongs and adapters, and sampling bottles. To avoid possible breakage and contamination, the air sampling train components, i.e., the filter and the Tenax®-GC tubes, as well as the Amberlite XAD-4 sampling tubes were all hand carried to the site by the sampling crew. Generators were rented at local dealers when electrical outlets were not available in the plant.

Because of the extensive sampling involved in the first two sites, i.e., Vulcan Materials Company and Stauffer Chemical Company, a four-man crew was required. The rest of the sampling trips were conducted by two- or three-man crews. Generally, 3 days were spent on each sampling site. The total number of samples analyzed for each sampling site is presented in Table 3. A summary of air sampling parameters for each site is given in Table 4. The sites at which HCBD breakthrough occurred are indicated. Breakthrough of HCBD was observed at 4,000 liters total volume of air sampled at a rate 3.5 liters/min. Breakthrough was not observed if the total volume was 2,000 liters or less. Detailed descriptions of the field sampling and presampling surveys conducted at each plant are presented in Appendix A.

Table 3. FIELD SAMPLING SUMMARY

Site	Air samples[a] (stations x train components x sampling period)	Total samples (number/type)
Vulcan	18 x 2 x 5	180 air
		10 soil
		4 water
Linden	No air samples	6 water
		3 solid
		1 soil
Stauffer	9 x 2 x 6	108 air
		5 soil
		6 water
		3 sediment
Dow	8 x 2 x 1	16 air
		3 soil
		1 water
Du Pont	8 x 2 x 1	10 air
		3 soil
		7 water
		3 sediment
Diamond Shamrock	8 x 3 x 1	24 air
		3 soil
		2 water
Ciba-Geigy	8 x 2 x 1	16 air
		4 soil
		2 water
Olin	8 x 3 x 1	24 air
		11 soil
		10 water
		1 sediment
PPG	10 x 3 x 1	30 air
		4 soil
		7 water
		3 sediment

---

[a] The total number of air samples consist of the number of air sampling stations times the components of the train, i.e., filter and Tenax®-GC resin times the number of sampling periods.

Table 4. SUMMARY OF AIR SAMPLING PARAMETERS

Plant	Average sampling vol. (ℓ)	Average sampling time (hr)	Rate (ℓ/min)	HCBD breakthrough
Vulcan[a]	(1) 150-200 (2) 800-1,000 (3) 4,000	(1) 1 (2) 4	0.5 0.5	No No
Stauffer[b]	450	2	3.5	No
Dow	4,100	20	3.5	Yes
Du Pont	4,200	21	3.5	Yes
Ciba-Geigy[c]	1,200-2,100	6-8	3.5	No
Diamond Shamrock	550	24	0.4	No
Olin[c]	1,500-2,000	9	3.5	No
PPG	1,200	24	0.4	No

---

[a] Five 4-hr periods.

[b] Six 4-hr periods (2 hr on, 2 hr off).

[c] Three 8-hr periods (each 2 to 3 hr sampling).

SECTION V

DISCUSSION OF RESULTS AND CONCLUSIONS

Air, soil, water, and sediment samples were collected from nine recommended industrial plants whose products included perchloroethylene, trichloroethylene, carbon tetrachloride, triazine herbicides, pentachloronitrobenzene and chlorine. In general, HCBD concentrations varied from a maximum, near the production and waste disposal areas, to a minimum, in the samples taken upwind. HCBD was detected as a vapor in every case. The results from each sampling site are discussed below.

VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Field sampling at Vulcan Materials Company's perchloroethylene plant at Wichita, Kansas, was conducted on May 20, 1975. Other compounds of interest produced at this plant include carbon tetrachloride and chlorine. The samples collected were: 180 air (90 filters and 90 Tenax®-GC columns), 10 solid, and 4 water.

Air Samples

The 180 air samples were collected from 13 samplers positioned upwind of the facility, nine samplers positioned immediately downwind of the general production and waste storage areas, and six additional samplers set further downwind beyond the northern plant boundary. The six samplers beyond the northern boundary were positioned at three locations with two samplers per location at 4 and 11 ft above ground, respectively. The upwind and farthest downwind samplers were operated continuously for a 4-hr period while those closer to the general production and waste storage area were operated only for the first hour of each 4-hr period. After each 4-hr period, the filter and the Tenax®-GC column in each sampler were replaced by fresh components. The sampling strategy was designed so that results of the analysis would elucidate: (a) the major sources and level of HCBD emission, (b) the diurnal and operation-related effects of HCBD emission, (c) the physical form, i.e., particulate or vapor of HCBD in the plant air, and (d) the variation of HCBD concentrations with respect to sampler distance above ground.

Sources and Levels of HCBD Emissions - The analytical data for the 180 air samples are tabulated in Table B-1 of Appendix B. Figure 4 shows the average concentrations of HCBD during the 20-hr sampling period at the 18 sampling stations. It is obvious that major sources of HCBD in the air are the production and waste storage ("Hex Pit") areas.

HCBD levels were highest, ranging up to 460 $\mu g/m^3$, in samples taken near the "Hex Pit;" the average HCBD concentration in that area was approximately 65 $\mu g/m^3$. The average HCBD concentration in all samples taken from the seven stations north of the plant boundary, downwind of the production and waste holding ("Hex Pit") areas, was approximately 8 $\mu g/m^3$. Only trace levels of HCBD were detected in upwind samples.

Variation of HCBD Emission with Time - The variation of HCBD levels over the 20-hr sampling period is shown in Figures 5 through 7. Figure 5 shows the levels of HCBD in the air samples immediately downwind of the production and waste storage areas. The air levels appear to reflect the plant activities, i.e., dumping of "hex residue." This is especially so at Stations 4 through 6 which are nearest the "Hex Pit."

The variation of HCBD levels in samples beyond the plant's northern boundary is shown in Figure 6. Each point is the average of two samplers positioned at different heights (4 and 11 ft). Again the figure indicates that the HCBD level increased at Stations 12 and 13 during hex dumping.

The low and rather constant HCBD levels detected in upwind samples during the entire sampling period as shown in Figure 7 indicate that the major source of HCBD is vapor from the "Hex Pit" and production plant and not from the buried wastes or plant area soil.

HCBD Concentrations Versus Sampler Heights - Samples were collected at two heights, 4 and 11 ft, simultaneously, at three locations for five successive time periods. Those results summarized in Table 5 demonstrate that there was no effect of sampler height.

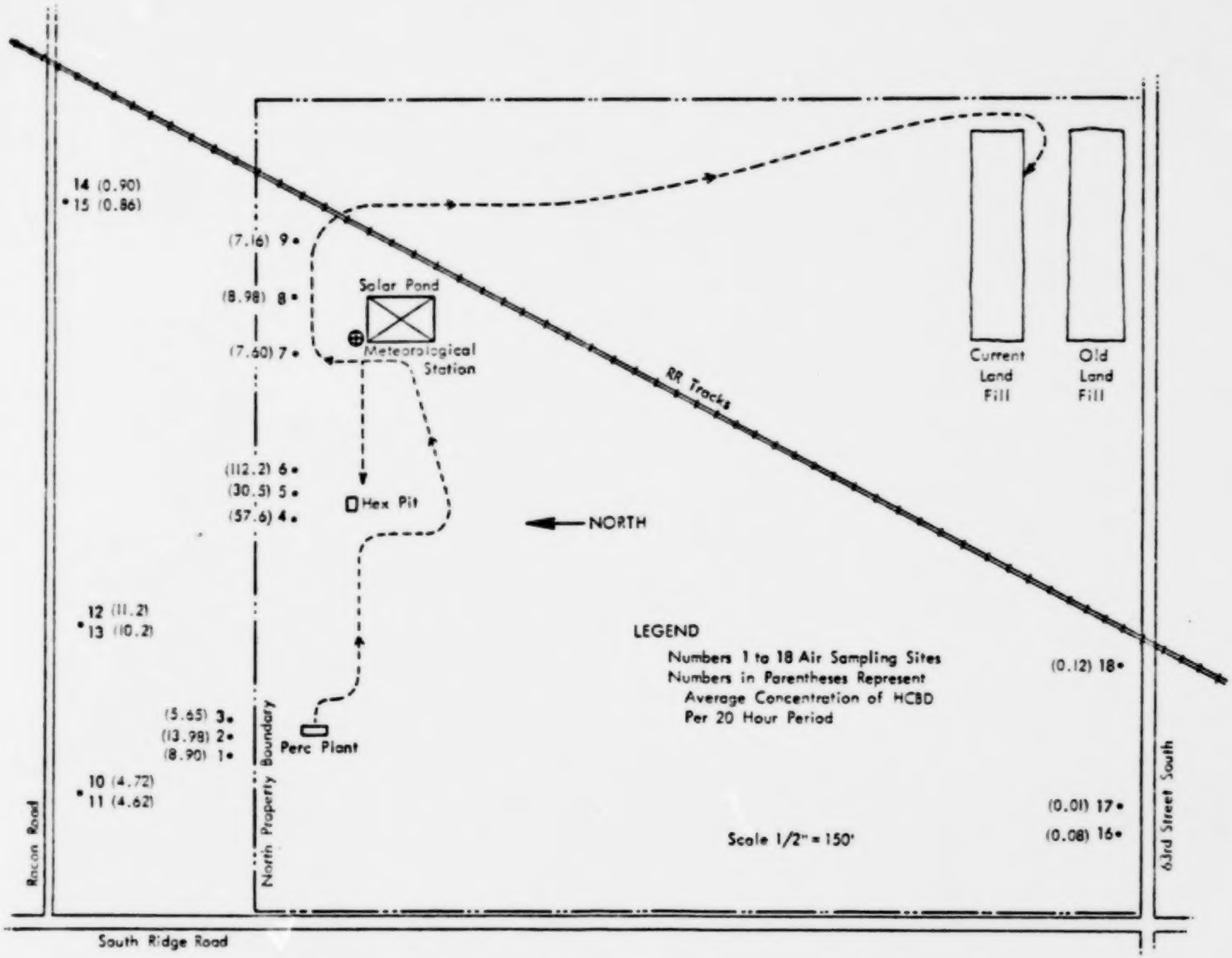

14 (0.90)
15 (0.86)
(7.16) 9
(8.98) 8
(7.60) 7
(112.2) 6
(30.5) 5
(57.6) 4
12 (11.2)
13 (10.2)
(5.65) 3
(13.98) 2
(8.90) 1
10 (4.72)
11 (4.62)
Racon Road
North Property Boundary
South Ridge Road
Solar Pond
Meteorological Station
Hex Pit
Perc Plant
NORTH
RR Tracks
Current Land Fill
Old Land Fill
LEGEND
Numbers 1 to 18 Air Sampling Sites
Numbers in Parentheses Represent Average Concentration of HCBD Per 20 Hour Period
Scale 1/2" = 150'
(0.12) 18
(0.01) 17
(0.08) 16
63rd Street South

Figure 4. Average concentrations ($\mu g/m^3$) of HCBD in air per 20-hr period at 18 sampling stations at Vulcan Materials Company, Wichita, Kansas

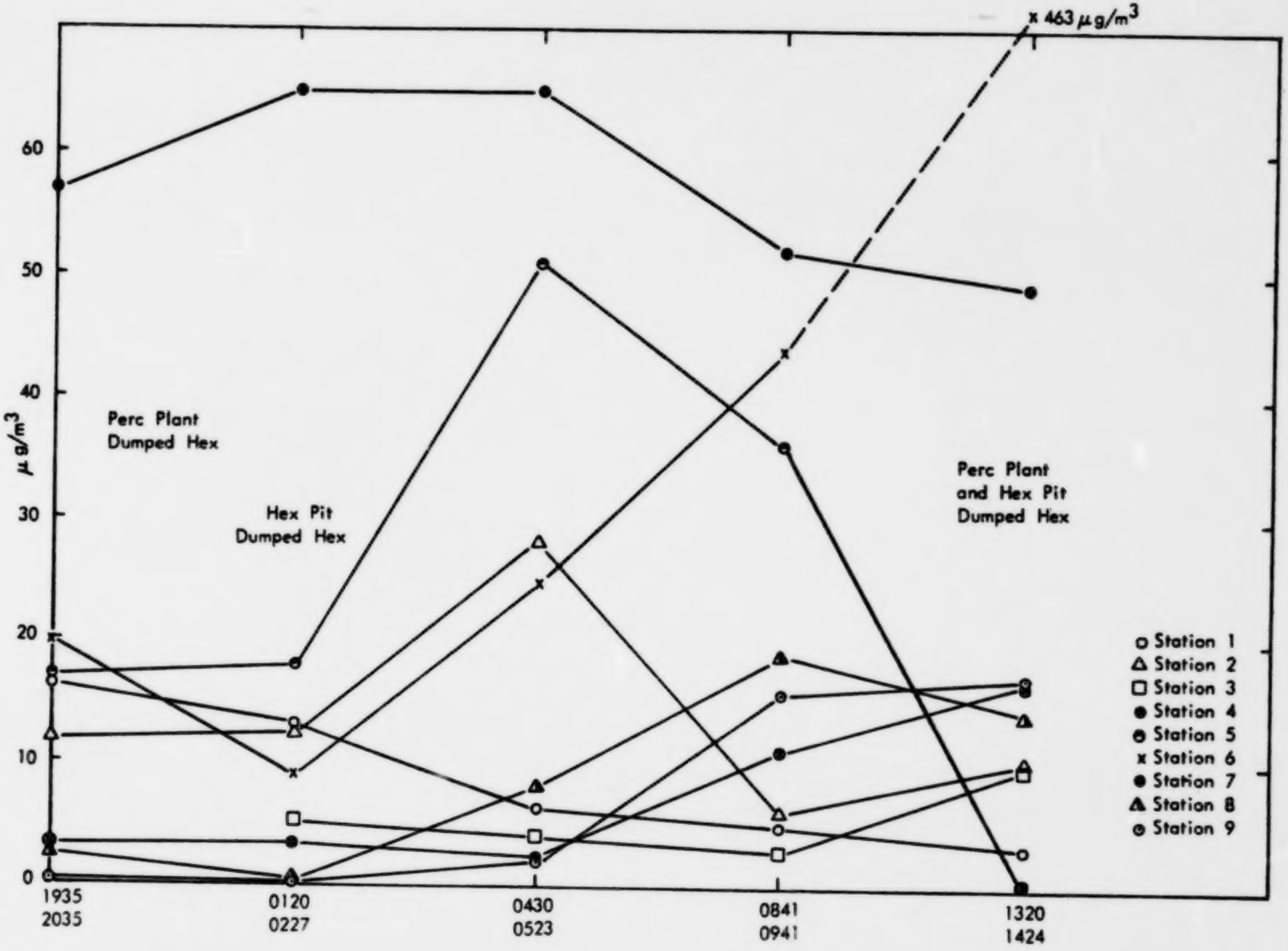


Figure 5. HCBD concentration per 20-hr period at sampling stations within the plant perimeter (Vulcan)

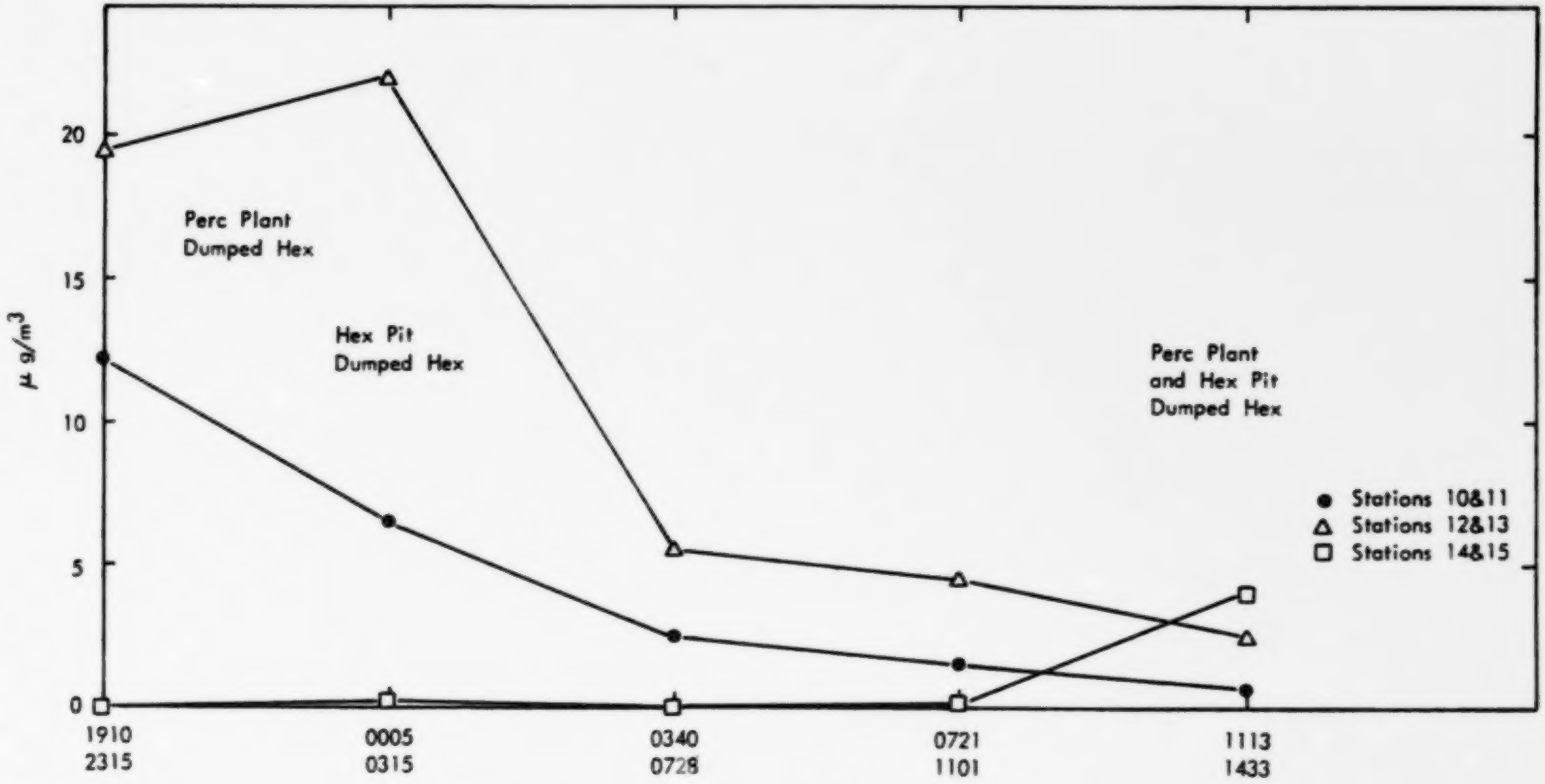


Figure 6. **HCBD concentration per 20-hr period, downwind** stations (Vulcan)

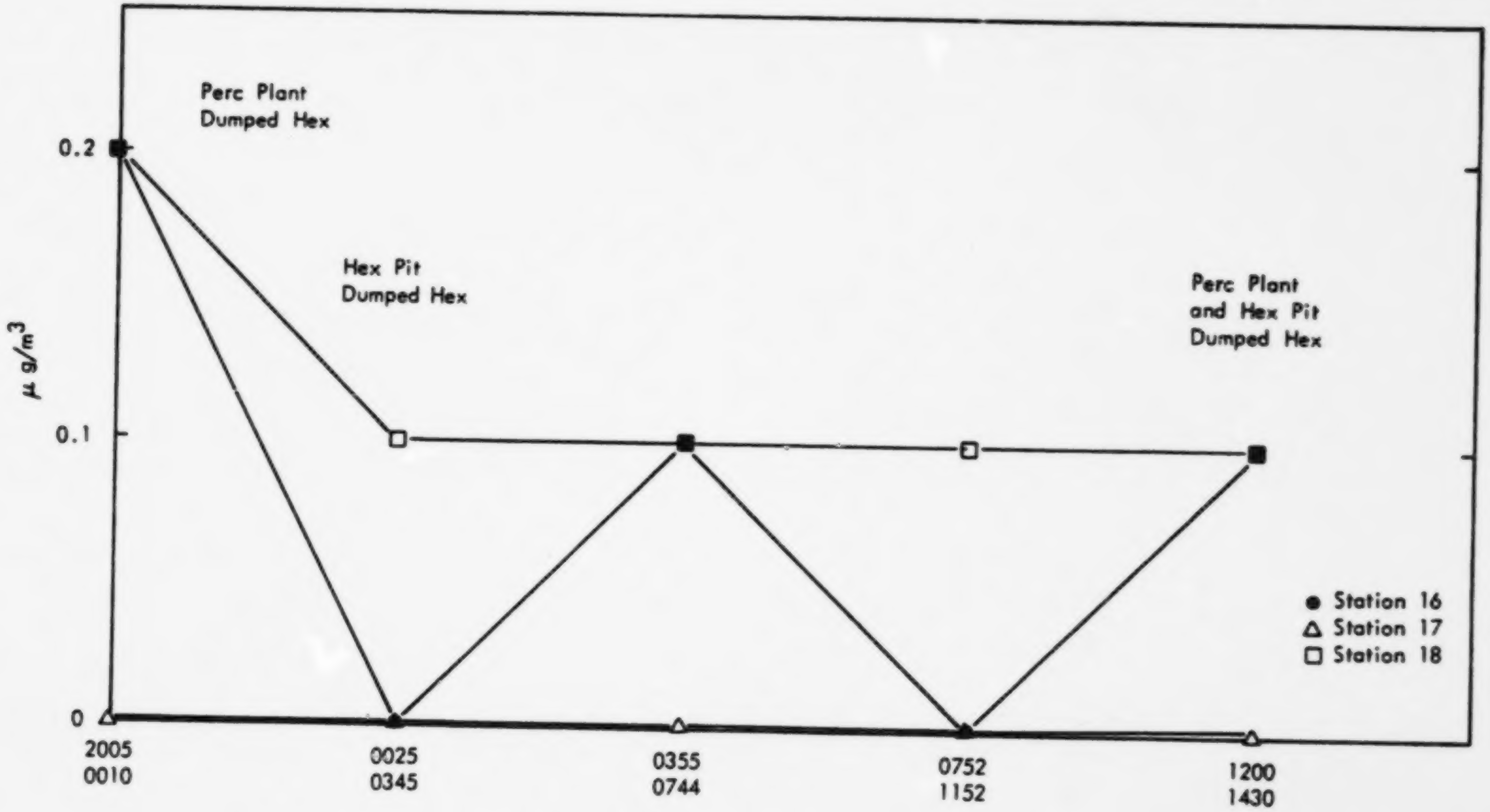


Figure 7. HCBD concentration per 20-hr period, upwind stations (Vulcan)

Table 5. COMPARISON OF HCBD LEVELS AT 4 AND 11 FT FOR FIVE TIME PERIODS

Station pairs	HCBD, $\mu g/m^3$ 4 ft	HCBD, $\mu g/m^3$ 11 ft
10 and 11	12.2	12.1
	6.5	6.5
	2.6	2.4
	1.6	1.4
	0.7	0.7
12 and 13	20	19
	22	22
	6	4
	5	4
	3	2
14 and 15	0.004	0.02
	0.2	0.2
	0.03	0.1
	0.1	0.2
	4.2	3.8

Precision of Sampling and Analysis - The agreement between each of the sample sets listed in Table 5 is excellent. If it is assumed that the HCBD levels were the same at the two sampler heights (4 and 11 ft), the results from each paired station, i.e., 10 and 11, 12 and 13, and 14 and 15, at each of the five sampling periods can be considered as duplicates and a pooled relative standard deviation (PRSD)* determined. The PRSD calculated from these data indicates the overall precision of the air sampling and analysis methods including sample collection, storage, recovery, and analysis. The PRSD was 14%. The PRSD is based on 13 duplicates for HCBD. Two duplicate measurements near the detection limit ($< 0.1\ \mu g/m^3$) were not included in the calculations.

Soil Samples

The nine soil samples and one "Hex Pit" solid sample were collected at the following locations: along the plant boundaries, transportation routes, landfill, the "Hex Pit" area and the production area (see Figure A-2). These sites were selected to determine HCBD soil levels associated with production waste disposal and transportation of wastes for disposal. Samples were also taken upwind and in adjacent agricultural fields to determine background concentrations in areas in the vicinity of the plant.

The results of the analysis of the nine soil and one solid samples, tabulated in Table 6, indicate that HCBD soil concentrations were generally in the high parts per billion range with the exception of the "Hex Pit" soil and the "Hex Pit" solids. HCBD is 0.1% in the "Hex Pit" soil and 10% in the "Hex Pit" solids.

Specifically, of the four plant boundaries, the highest level of HCBD, 0.106 ppm, was observed in soil from the southern boundary (S-8). On the other three boundaries, levels of the two substances ranged from 0.005 to 0.030 ppm for HCBD.

The observation of relatively high concentrations of HCBD in the southern boundary soil sample (S-8) could very well be the source of HCBD in the air samples collected at Air Sampling Stations 16 through 18. The high concentrations in upwind soil samples (S-8) could be due to the nearby landfill. This rationale is supported by the presence of 0.36 ppm of HCBD found in the S-5 sample, which indicates the landfill is a source of surface contamination in the immediate area.

---

* The PRSD was calculated as follows:

$$S = \sqrt{\sum_{i=1}^{n} (X-\overline{X})^2/0.889}$$

$$RSD = S/\overline{X} \times 100$$

$$PRSD = \sqrt{\sum_{i=1}^{n} RSDi^2/n}$$

Table 6. HCBD CONCENTRATIONS IN SOIL AND SEDIMENT FROM VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Samples[a/]	Sample weight (g)	Concentration (μg/g) HCBD
S-2	42.8	2.28
S-3	2.5	980
S-4	48.4	0.22
S-5	38.7	0.36
S-6	40.5	0.049
S-7	29.6	0.030
S-8	35.6	0.106
S-9	43.5	0.018
S-10	34.2	0.005
"Hex Pit" solids	0.95	10%
Control		ND

---

a/ S-2 - Route from "Perc Plant" to "Hex Pit."
S-3 - "Hex Pit."
S-4 - Route from "Hex Pit" to landfill.
S-5 - Landfill (60 yards north of 63rd Street and 0.4 miles east of Ridge Road.
S-6 - Landfill (180 yards north of 63rd Street and 0.4 miles east of Ridge Road.
S-7 - Landfill (Ridge Road to telephone pole).
S-8 - Upwind.
S-9 - Downwind.
S-10 - Western boundary (cornfield).
Control - Soxhlet apparatus.

From 0.22 to 2.2 ppm of HCBD were observed in the soil on the route to the "Hex Pit" (S-2) and in the soil from the "Hex Pit" to the landfill (S-4). The high concentrations of HCBD found in the "Hex Pit" solids were expected since the "hex residues" consist mainly of HCB and HCBD.[3] Results of the analyses of air samples collected at Air Sampling Stations 4, 5, and 6 showed consistently high concentrations of HCBD (see Figure 5). Furthermore, the HCBD levels found in the water layer covering the "Hex Pit" were also relatively high. The results of the water analysis are presented below.

Water Samples

Two samples were taken from Cowskin Creek (Figure A-3) which receives water from the sanitary sewer system and plant heat exchangers. Samples were taken from the "Hex Pit" and "Solar Pond" to determine their contribution to HCBD levels in air and into the deep well which receives water from the "Solar Pond."

The results of the analyses are shown in Table 7. No HCBD was detected in Cowskin Creek. The presence of a high level of HCBD (231 ppb) in the "Hex Pit" water is expected since this water was used to cover the "hex residues" dumped in the pit. This water should be saturated with HCBD. The level of HCBD in the "Solar Pond" is two orders of magnitude lower than that in the "Hex Pit" water. The source of HCBD in the "Solar Pond" water could be from leaching of the soil or from vapor or airborne particulate from the neighboring "Hex Pit."

Plant Summary

The results of the analysis of all air, soil, and water samples indicate that the "Hex Pit" is the source of the highest levels of HCBD. All of the HCBD found in the air was as the vapor; no particulate HCBD was observed. Variations in the air levels at the downwind stations were related to the dumping of solid "hex residues." HCBD concentrations in soil (excluding the "Hex Pit" area) ranged from 0.005 ppm to over 2 ppm. The water samples taken from the "Hex Pit" and "Solar Pond" contained 231 and 2.2 ppm HCBD, respectively. No HCBD was detected in Cowskin Creek.

LINDEN CHLORINE PLANT, LINDEN, NEW JERSEY

The survey of Linden Chlorine Plant was conducted on May 29, 1975. During the intended presampling survey, it was apparent that an extended air sampling plan was not warranted. Therefore, sediment, water, and soil samples were taken during the survey.

Table 7. HCBD CONCENTRATIONS IN WATER FROM VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Samples	Volume sampled (liter)	Concentration (μg/ℓ) HCBD
"Hex Pit" water	0.315	231
Solar Pond	0.335	2.2
Upstream (Cowskin Creek)	323	ND
Downstream (Cowskin Creek)	365	ND

ND - None detected.

The Linden Plant was selected as a tentative sampling site because graphite electrodes were used in the production of chlorine and the plant produced a single product. During our visit we learned that graphite electrodes had been phased out at the end of March 1975.

Seven water and four solid samples were analyzed for HCBD. The results are listed in Table 8. The results indicate that parts per trillion levels of HCBD were present in the holding pond (inlet and outlet), spent brine, and the upstream and downstream water of the plant. No HCBD was found in the tap and process water.

Three of the four solid samples contained HCBD at a level of 0.04 to 0.18 μg/g. The highest level of HCBD, 0.18 μg/g, was observed in the waste stream sludge downstream of the plant.

Due to the complexity, i.e., large number of peaks, of these chromatograms, selected samples were fortified with standards to confirm the presence of HCBD. In addition, the samples were prepared and analyzed in duplicate and/or in triplicate.

Plant Summary

Air samples were not taken at the Linden Chlorine Plant because graphite electrodes had been phased out prior to our sampling. Water samples contained low levels of HCBD (0.02 to 0.05 μg/liter). The highest concentration of HCBD (0.18 μg/g) was found in the sludge taken from the waste downstream of the plant. The level of HCBD detected in the water and solid samples indicate this plant is not a current source of significant quantities of this material.

STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Field sampling at Stauffer Chemical Company's perchloroethylene plant at Louisville, Kentucky, was conducted on June 12, 1975. A total of 108 air, (54 filters and 54 Tenax®-GC columns) 5 soil, 3 sediment, and 6 water samples was collected. The results of the analysis of these samples for HCBD are discussed below.

Air Samples

The 108 air samples were collected from nine samplers positioned at nine locations surrounding the plant. Because the entire western and part of the northern boundaries of the plant are surrounded by a flood wall along the Ohio River, the positioning of downwind samplers was limited.

Table 8. HCBD CONCENTRATIONS IN WATER AND SOLIDS FROM LINDEN CHLORINE COMPANY, LINDEN, NEW JERSEY

Sample	Concentration (μg/ℓ) HCBD
Water	
Holding pond, inlet	0.04
Holding pond, outlet	0.04
GAF weir, upstream of Cl_2 plant	0.05
Waste stream, downstream of Cl_2 plant	0.02
Process water	ND
Tap water	ND
Spent brine water	0.08
Solid	Concentration (μg/g)
Holding pond, settled and suspended	0.04
Dredged solids adjacent to holding pond	0.09
Waste stream, downstream of Cl_2 plant	0.18
Soil, around one of the cell buildings	ND

Sampling at these nine locations was divided into six 4-hr periods and all samplers were operated 2 hr of each 4-hr period. After each 4-hr period, the filters and the Tenax®-GC columns in the samplers were replaced. The sampling was conducted so that the analytical results would indicate (a) the sources and levels of HCBD, (b) the diurnal and plant operational effects, if any, and (c) the physical form of the substance in the air.

Sources and Levels of HCBD - The results of the analyses are listed in Table B-2 of Appendix B. The filters collected at the same sampling station at various sampling times were combined to form one sample. The combined filter analysis remains indicative of the specific form of HCBD present in the plant air while reducing the number of filters to be analyzed from 54 to nine. The average HCBD concentration per 24-hr period at each sampling station is shown in Figure 8.

The average HCBD concentrations at upwind Stations 1 and 2 were 0.03 $\mu g/m^3$, while at the downwind stations (3 through 9), HCBD concentrations were from 0.06 to 5.8 $\mu g/m^3$. Of the downwind stations, the highest levels of HCBD were observed at Stations 4 and 5 which were located downwind from the perchloroethylene-carbon tetrachloride plant. The level of HCBD was somewhat lower at Station 6, which was further downwind from the perchloroethylene-carbon tetrachloride plant. All HCBD was found in the vapor form.

Variation of HCBD Emissions with Time - HCBD levels detected during the 24-hr sampling period for each of the nine sampling stations are plotted versus sampling time in Figure 9.

The diurnal effect on HCBD levels was indistinct. Only slightly lower levels were observed for the downwind stations during the early morning hours.

The removal of the solid waste drums from the plant area occurred during the latter part of the first sampling period and early part of the second (1400 to 1500 hr). Levels of HCBD peaked during the third sampling period at downwind Stations 5 and 6. The exact time when the drums were removed from the drum loading area and transported off-site was not known.

Soil and Sediment Samples

Five soil samples were collected from the soil along the plant boundaries near the hex storage facilities and along the waste transportation route. Two sediment samples were taken from the Ohio River

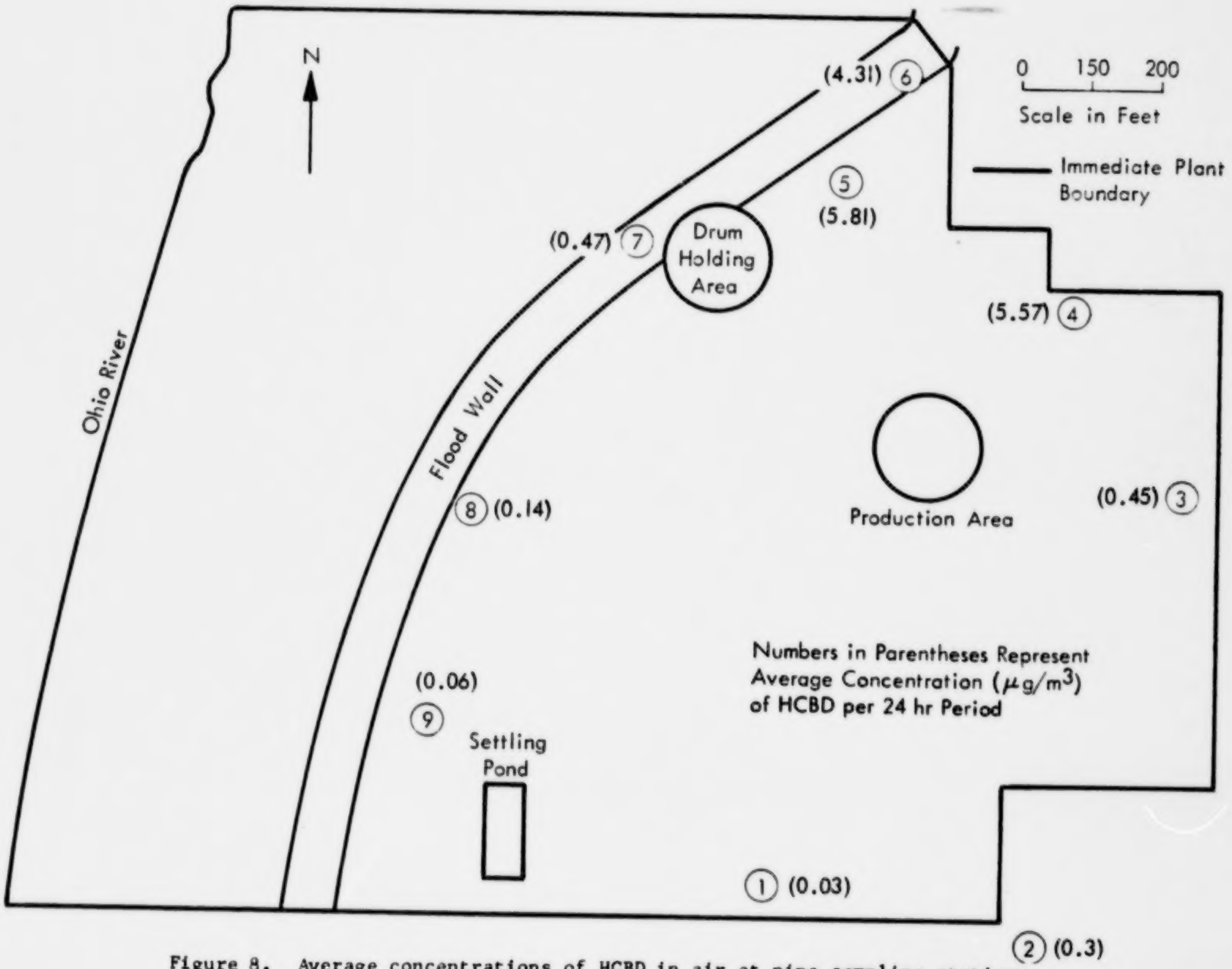

N
0 150 200
Scale in Feet
Immediate Plant Boundary
Ohio River
Flood Wall
(4.31) ⑥
⑤ (5.81)
(0.47) ⑦
Drum Holding Area
(5.57) ④
Production Area
(0.45) ③
⑧ (0.14)
Numbers in Parentheses Represent Average Concentration ($\mu g/m^3$) of HCBD per 24 hr Period
(0.06) ⑨
Settling Pond
① (0.03)
② (0.3)

Figure 8. Average concentrations of HCBD in air at nine sampling stations at Stauffer Chemical Company, Louisville, Kentucky

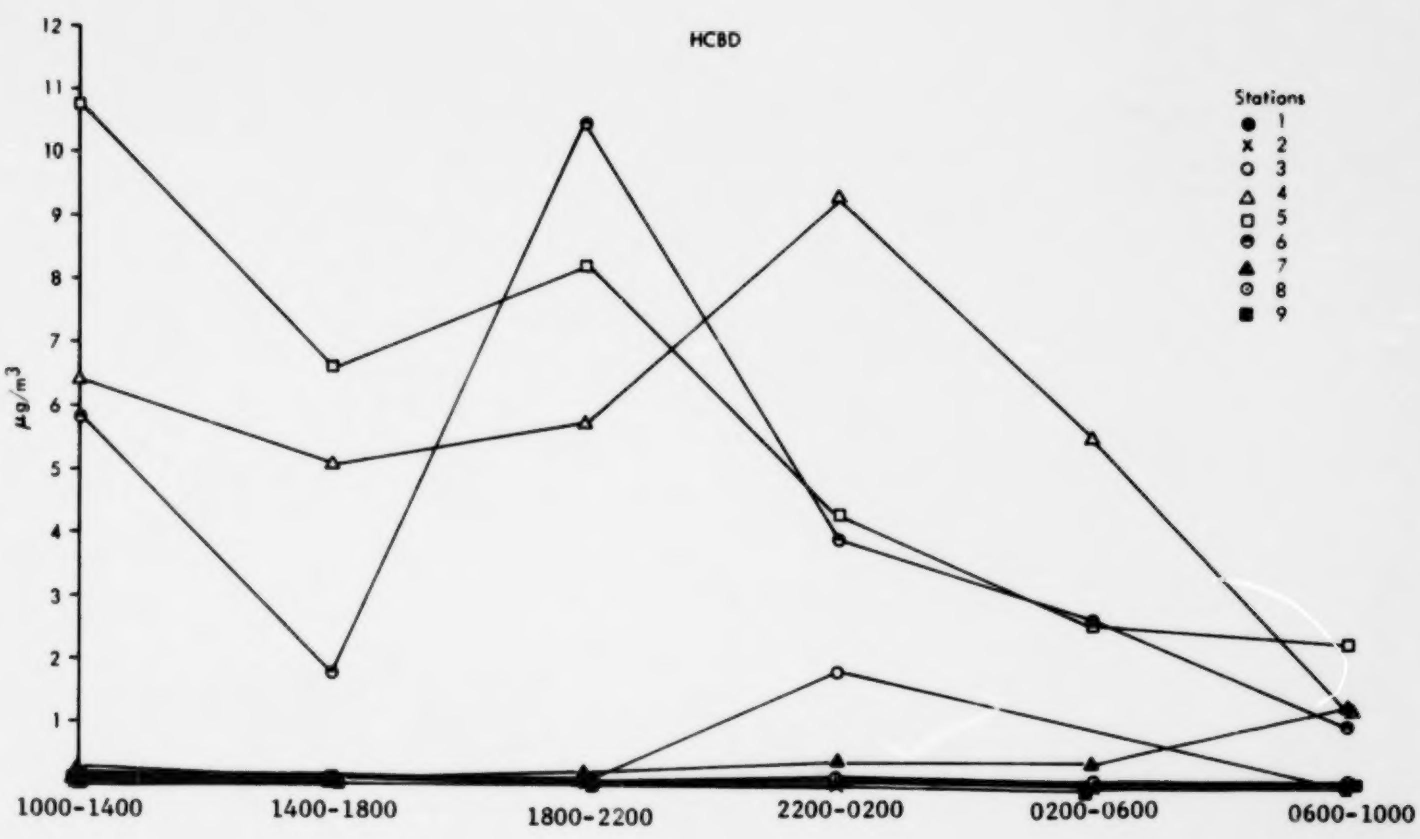


Figure 9. HCBD concentration per 24-hr period at sampling stations around the plant (Stauffer)

and one was taken from the holding pond. The results, shown in Table 9, indicate that the soil and sediment levels were from 1 to 30 ppb except for the soil at the drum loading area (28.5 ppb), and the sediment from the holding pond (26 ppb). The soil concentrations follow the same general pattern as the air concentrations, i.e., the upwind sample had the least amount of HCBD, 0.001 μg/g, while the downwind sample (northern plant boundary) contained 0.03 μg/g HCBD. The soil samples from the main road and the settling pond area had HCBD levels between the downwind and upwind levels. The soil in the drum loading area which contained 28.5 μg/g HCBD was obviously contaminated during waste handling operations.

Of the three sediment samples analyzed, the settling pond sample contained the highest levels of HCBD, 26 μg/g. Sediments collected at the Ohio River, both up and downstream, were in the parts per billion range.

The higher level of HCBD in the upstream sample compared to the downstream sample is probably due to contamination from sampling or sample preparation. Both HCBD sediment values however are near the detection limit where the relative error in analysis is high.

Water Samples

The six water samples were collected from the plant well and the settling pond.

The results, listed in Table 10, indicate that HCBD was present in the plant well water at 0.1 μg/liter. Analysis of the grab inlet and outlet water samples from the settling pond shows that the settling pond treatment has a negligible effect on HCBD concentration. The XAD-4 results agree quite closely with the results obtained for the outlet grab sample. The comparison served as a check on the trapping efficiency and recovery for the XAD-4 system in a real waste stream. However, data from the 24-hr composite sample (collected by Stauffer) indicate no HCBD was present in both inlet and outlet samples.

The differences in results observed in "grab" versus 24-hr composite samples imply that sampling time as well as sampling technique is important for HCBD. Generally, the 24-hr composite sample is more representative. However, in the samples analyzed here, it is very likely that HCBD was lost during sampling for the 24-hr composite because of the relatively high volatility of HCBD. Furthermore, the agreement between results obtained for the Amberlite XAD-4 resin and the grab sample substantiate their validity.

Table 9. HCBD CONCENTRATIONS IN SOIL AND SEDIMENT FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Samples	Concentration (µg/g) HCBD
Soil	
S-1 - Upwind (southern plant boundary)	0.001
S-2 - Plant road	0.006
S-3 - Drum loading area	28.5
S-4 - Downwind (northern plant boundary)	0.03
S-5 - Settling pond area	0.005
Sediment	
R-1 - Settling pond	26
R-2 - Ohio River (upstream)	0.017
R-3 - Ohio River (downstream)	0.002

Table 10. HCBD CONCENTRATION IN WATER FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Samples	Concentration (µg/ℓ) HCBD
Plant well water	0.1
Settling pond inlet (grab)	25
Settling pond outlet (grab)	23
Settling pond inlet (24-hr composite)	ND
Settling pound outlet (24-hr composite)	ND
Settling pond outlet (Amberlite XAD-4)	21

Plant Summary

The results of the analyses of all air samples indicate that the carbon tetrachloride-perchloroethylene plant is the major source of HCBD in the general plant area. No significant diurnal variation was observed. All of the detected HCBD was present in the vapor form.

The highest concentration of HCBD in soil (28.5 μg/g) was near the "drum loading area." This level indicates a localized contamination from solid waste handling. Otherwise, HCBD levels ranged from 0.001 to 0.03 μg/g in other soil samples around the plant.

A sediment sample from the settling pond showed a high level of HCBD, 26 μg/g, but levels observed in samples taken from the Ohio River were insignificant.

Results for grab samples collected at the settling pond outlet show excellent agreement with samples collected through Amberlite XAD-4 resin, and 23 versus 21 μg/liter of HCBD.

DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Field sampling at Dow Chemical Company's carbon tetrachloride-perchloroethylene plant at Pittsburg, California, was conducted on August 7, 1975. A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 3 soil, and 1 water samples was collected.

Air Samples

The 24 air samples were collected from 8 samplers, 2 of which were placed upwind at the western plant boundary, 3 at the midplant area, immediately downwind from the production and incinerator areas, and 3 farther downwind at the eastern plant boundary. Because no significant diurnal effects were observed at the Vulcan and Stauffer plants where air sampling was conducted in discrete 4-hr periods, the sampling at Dow was conducted for an integrated 24-hr period. Each sampling train was set up with two Tenax®-GC columns in tandem to check for breakthrough of HCBD. The samplers were positioned so that results of the analysis would indicate (a) the source and level of HCBD emission, (b) the physical form of HCBD, and (c) the efficiency of HCBD collection.

Sources and Levels of HCBD - The results are presented in Table B-3 of Appendix B. Average HCBD concentrations are shown in Figure 10. HCBD

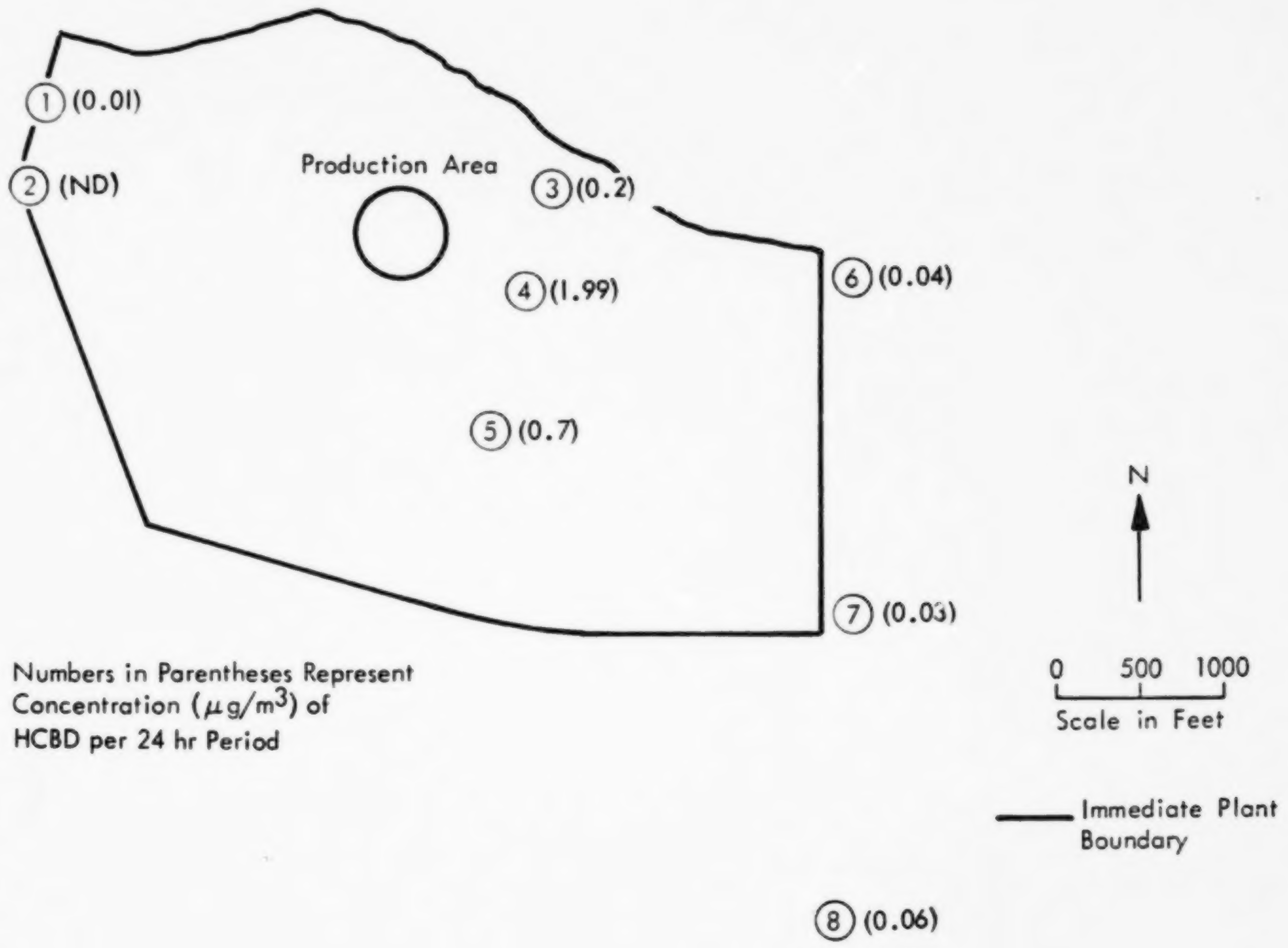

① (0.01)
② (ND)
Production Area
③ (0.2)
④ (1.99)
⑤ (0.7)
⑥ (0.04)
⑦ (0.03)
⑧ (0.06)
N
0 500 1000
Scale in Feet
Immediate Plant Boundary
Numbers in Parentheses Represent Concentration ($\mu g/m^3$) of HCBD per 24 hr Period

Figure 10. Average concentrations of HCBD in air at eight sampling stations at Dow Chemical Company, Pittsburg, California

concentrations in the upwind samples were from nondetectable to less than 0.01 μg/m^3, while the downwind samples showed concentrations from 0.04 to 2.0 μg/m^3. The maximum concentration of HCBD (2.0 μg/m^3) was detected at Station 4 which was located east of the plant area. The recorded wind direction during the sampling was primarily from the west. Station 1 directly upwind of the plant area contained 0.01 μg/m^3 HCBD.

The high concentrations at Stations 3 through 5 indicate that the source of emission is localized and centered in the production area which includes the thermal oxidizer. The remaining results show that there was no widespread contamination of the plant. All HCBD was found in the vapor form.

HCBD Collection Efficiency - The detection of HCBD in the back-up Tenax®-GC tube indicates that HCBD, at 24-hr continuous sampling time with 4,000 liters sampled, passed through the first tube. It is believed that HCBD first loaded the front tube and was gradually displaced and transported into the back-up tube. This speculation is based upon the data observed at Station 7 where the sample was collected at a slower rate resulting in a lower volume of air sampled (962 liters). At this station, 0.3 μg/m^3 HCBD was found in the first tube and none in the back-up tube. Based on these results, air sample volumes were reduced for 24-hr integrated air samples taken subsequent to these analyses.

Soil Samples

Three soil samples were collected along the eastern, western, and southern plant boundaries.

The results shown in Table 11 indicate that HCBD was not detected in the western and eastern plant boundary soil, while the southern boundary contained a very low concentration of 0.005 μg/g.

Water Sample

One water sample was collected from the New York slough at the northwest corner of the plant. However, the sample was lost when the bottle containing the water was broken during shipping.

Plant Summary

The results of the analysis of air samples from the Dow plant indicate that the chlorinated hydrocarbon plant area which includes the carbon tetrachloride, tri- and perchloroethylene plants is a source of HCBD. HCBD levels are lower at the upwind western boundary (< 0.01 μg/m^3) than at downwind locations (0.02 to 0.3 μg/m^3) indicating that the source of HCBD is vapor from the plant area.

Table 11. HCBD CONCENTRATIONS IN SOIL FROM DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Samples	Concentration (μg/g) HCBD
Western plant boundary	ND
Eastern plant boundary	ND
Southern plant boundary	0.005

The absence of HCBD in the millipore filter indicates that HCBD is in the form of vapor rather than particulates in the plant air.

HCBD was found only in the soil collected along the southern plant boundary.

E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Field sampling of Du Pont's carbon tetrachloride plant at Corpus Christi, Texas, was conducted on August 3, 1975. Because of generator failure, only five air sampling stations were in operation, which resulted in the collection of a total of 15 air samples. The generator failure limited the location as well as the number of samples taken. In addition to the air samples, 6 soil and sediment, and 7 water samples were collected.

Air Samples

The 15 air samples were collected from five samplers of which two were positioned upwind, and three were positioned downwind from the general production area. The samplers were operated continuously for a period of 24 hr, with two Tenax®-GC columns in tandem.

Sources and Levels of HCBD - The results of the analysis are listed in Table B-4 of Appendix B and shown in Figure 11. The average concentrations of HCBD at upwind Stations 1 and 2 ranged from 0.003 to 0.22 $\mu g/m^3$, while downwind Stations 3 through 5 showed concentrations from 0.027 to 0.034 $\mu g/m^3$. The HCBD was present entirely as a vapor which agreed with previous sampling results.

HCBD Breakthrough - The detection of HCBD in the back-up Tenax®-GC tube indicates that HCBD, sampled under conditions similar to those used at Dow's Pittsburg, California, plant (24-hr sampling period and 4,000 liters sampled), passed through the first tube. As a result, lower air flow rates were used for subsequent air sampling.

Soil and Sediment Samples

The three soil samples were collected along the southern and northern plant boundaries, and near the landfill site. The sediment samples were collected at the storm runoff outfall, settling pond inlet and outlet. Results of analysis are listed in Table 12.

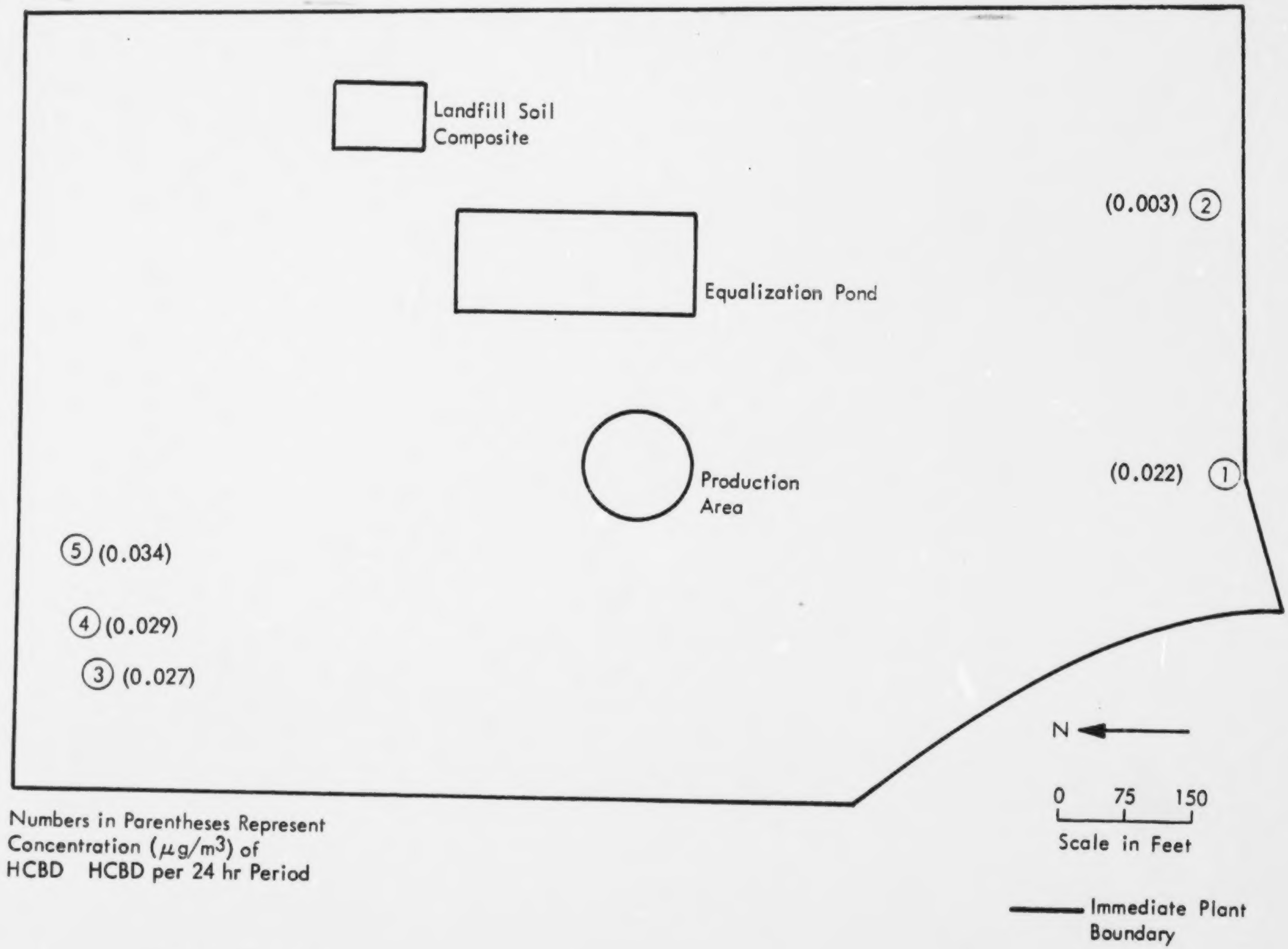

Landfill Soil Composite
Equalization Pond
(0.003) ②
Production Area
(0.022) ①
⑤ (0.034)
④ (0.029)
③ (0.027)
N
0 75 150
Scale in Feet
Numbers in Parentheses Represent Concentration (µg/m³) of HCBD HCBD per 24 hr Period
Immediate Plant Boundary

Figure 11. Average concentrations of HCBD in air at five sampling stations at E. I. du Pont de Nemours Company, Corpus Christi, Texas

Table 12. HCBD CONCENTRATIONS IN SOIL AND SEDIMENT FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

Soil samples	Concentration (µg/g) HCBD
Upwind (southern boundary)	ND
Downwind (northern boundary)	ND
Landfill area	0.004
Sediment	
Settling pond inlet	ND
Settling pond outlet	ND
Storm runoff outfall	0.061

HCBD was not detected in either the upwind or downwind soil samples. HCBD concentration in the landfill sample was 0.004 μg/g.

In the sediment, HCBD was detected only in the storm runoff outfall sample at 0.061 μg/g. No HCBD was detected in either the inlet or outlet settling pond sediments.

Water Samples

The seven water samples were collected from plant facilities where water was used either during the production process or for cleanup following production. Two types of sampling were conducted at the settling pond, grab sampling and sampling through an Amberlite XAD-4 column.

The results of the analysis of the seven samples are listed in Table 13. HCBD was detected only in the water standing in the landfill at 0.319 μg/liter HCBD.

Plant Summary

The slightly elevated levels of HCBD in the downwind air samples indicate that the production area is a source of HCBD emission.

The concentration of HCBD was highest in the soil collected around the landfill area. Of the water samples, HCBD was detected only in the landfill standing water.

In general, the levels of HCBD in this plant were very low. The plant began operations as recently as 1973 and appears to be successful in minimizing HCBD emissions.

DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Field sampling at Diamond Shamrock Corporation's trichloroethylene plant at Deer Park, Texas, was conducted on August 20, 1975. A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 3 soil, and 2 water samples was collected.

Air Samples

The 24 air samples were collected from eight sampling stations, five of which were positioned in a circle around the production area; two were located along the north boundary and one was at the south boundary of the plant area. Changing wind directions (Table A-13) during sampling prevented the samplers from being positioned in upwind and downwind locations. The sampling was conducted over three 8-hr sampling periods covering 3 days. Rain interrupted the second sampling

Table 13. HCBD CONCENTRATIONS IN WATER FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

Samples		Concentration (μg/ℓ) HCBD
W-1	Raw plant water before use	ND
W-2	Settling pond inlet (amberlite)	ND
W-3	Settling pond inlet (grab)	ND
W-4	Settling pond outlet (amberlite)	ND
W-5	Settling pond outlet (grab)	ND
W-6	Storm runoff outfall (grab)	ND
W-7	Water standing in landfill	0.32

period and prevented sampling during 1200 to 1700 hr on any of the 3 days. The samplers were operated at 0.4 liters/min, resulting in 600 liters or less of air sampled. Two Tenax®-GC columns were used in tandem.

Source and Levels of HCBD - The results of the analysis are listed in Table B-5 in Appendix B. A simplified plant map with the sampling locations and the 24-hr average concentrations is shown in Figure 12.

HCBD was detected in concentrations ranging from 0.09 to 2.4 μg/m^3. The highest concentration of HCBD was detected at the southern plant boundary. The next highest concentration of HCBD, 1.80 μg/m^3, was detected at Station 5 which is located immediately north of the general production area. The remaining samples were of similar concentration and in the 0.1 to 0.2 μg/m^3 range. All HCBD was detected as a vapor. There was no pattern of upwind-downwind concentrations because of variable wind direction during the sampling.

Collection Efficiency - The HCBD was trapped entirely by the front Tenax®-GC column. The breakthrough observed in previous sampling at Dow and Du Pont was eliminated by using a smaller critical orifice (26 gauge needle) that resulted in an air flow of 0.4 liters/min.

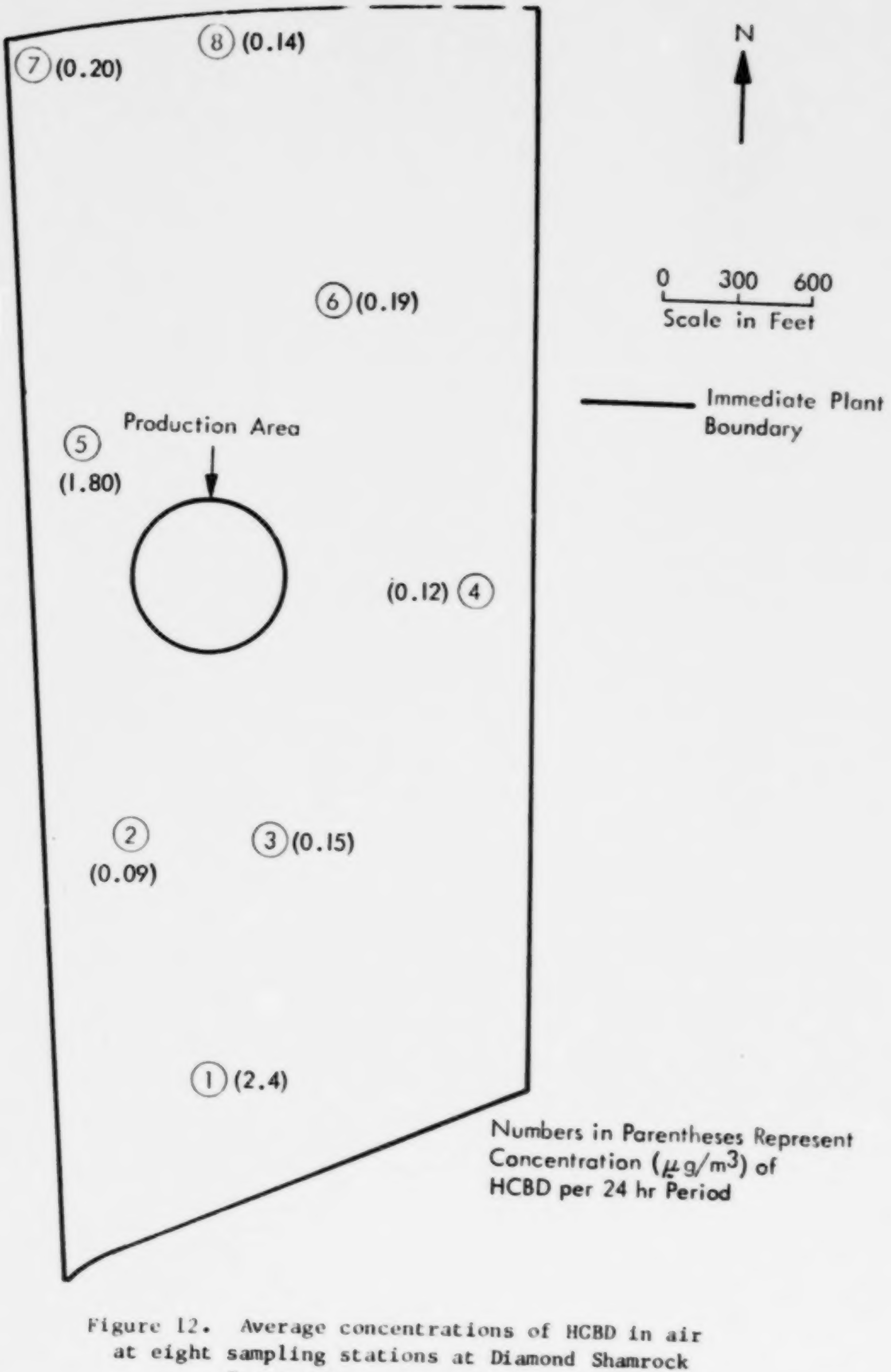
Soil Samples

Three soil samples were collected from along the northern plant boundary, along the southern plant boundary, and the production area.

The results of the analysis of the three soil samples are shown in Table 14. The data indicate that the highest concentrations of HCBD (19 μg/g) was detected in the production area. A higher level of HCBD was detected in the soil sample collected near Stations 7 and 8. These levels coincide with the fact that the HCBD concentration at Air Sampling Station 1 was higher than those observed at Stations 7 and 8. The source of the relatively higher levels of HCBD in air and soil at the southern boundary is not known.

Water Samples

Two water samples collected from the raw plant water and plant effluent for the analysis of HCBD. The results shown in Table 15 indicate that no HCBD was detected in the raw water. However, 2.0 μg/liter of HCBD was detected in the plant effluent. This plant effluent is discharged into nearby Patrick Bayou.



⑦ (0.20)
⑧ (0.14)
N
⑥ (0.19)
0 300 600
Scale in Feet
Immediate Plant Boundary
Production Area
⑤ (1.80)
(0.12) ④
② (0.09)
③ (0.15)
① (2.4)
Numbers in Parentheses Represent Concentration ($\mu g/m^3$) of HCBD per 24 hr Period

Figure 12. Average concentrations of HCBD in air at eight sampling stations at Diamond Shamrock Corporation, Deer Park, Texas

Table 14. HCBD CONCENTRATIONS IN SOIL FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Soil samples	Concentration (μg/g) HCBD
Upwind (southern boundary)	0.15
Downwind (northern boundary)	0.057
Production area	19

Table 15. HCBD CONCENTRATIONS IN WATER FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Samples		Concentration (μg/ℓ) HCBD
W-1	Raw unused plant water	ND
W-2	Plant effluent	2.0

Plant Summary

The level of HCBD in the air ranged from 0.09 to 2.4 μg/m^3. The highest levels were at the southern plant boundary and directly north of the production area. HCBD was detected solely as a vapor. No breakthrough occurred during the sampling.

HCBD was detected in soil samples collected along the northern and southern plant boundaries at parts per billion levels. However, in the production area, 19 ppb of HCBD was found.

The inlet plant water did not contain detectable quantities of HCBD while the process plant effluent showed HCBD at 2.0 μg/liter.

CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Field sampling at Ciba-Geigy Corporation's triazine herbicide plant in St. Gabriel, Louisiana, was conducted on August 13, 1975. A total of 16 air, 4 soil, and 2 water samples was collected.

Air Samples

The 16 air samples were collected from eight samplers which surrounded the entire production area. Stations 2, 4, 6, and 8 were positioned about 400 ft, and Stations 1, 3, 5, and 7 were approximately 1,000 ft, from the production area. One Tenax®-GC column was used in the sampling train. To avoid possible breakthrough, the sampling was conducted 2 hr of every 8-hr period, over a 24-hr period.

Source and Levels of HCBD - The results of the analyses are listed in Table B-6 of Appendix B. The average concentrations of HCBD are shown for each sampling station in Figure 13. HCBD concentrations were from nondetectable to 0.1 μg/m^3. The wind directions were quite erratic during sampling, and upwind-downwind patterns were not observed. The relatively high HCBD level (0.1 μg/m^3) observed at Station 8 is anomalous, because at no time during the sampling was the wind from the east. Therefore it is possible that this sample could have been contaminated--either in the plant during sampling or in the laboratory during sample preparation. This explanation is further substantiated by the absence of HCBD in the sample collected at Station 7 which was positioned south of Station 8 and southwest of the production area.

All HCBD in the plant air was in the form of vapor rather than particulates.

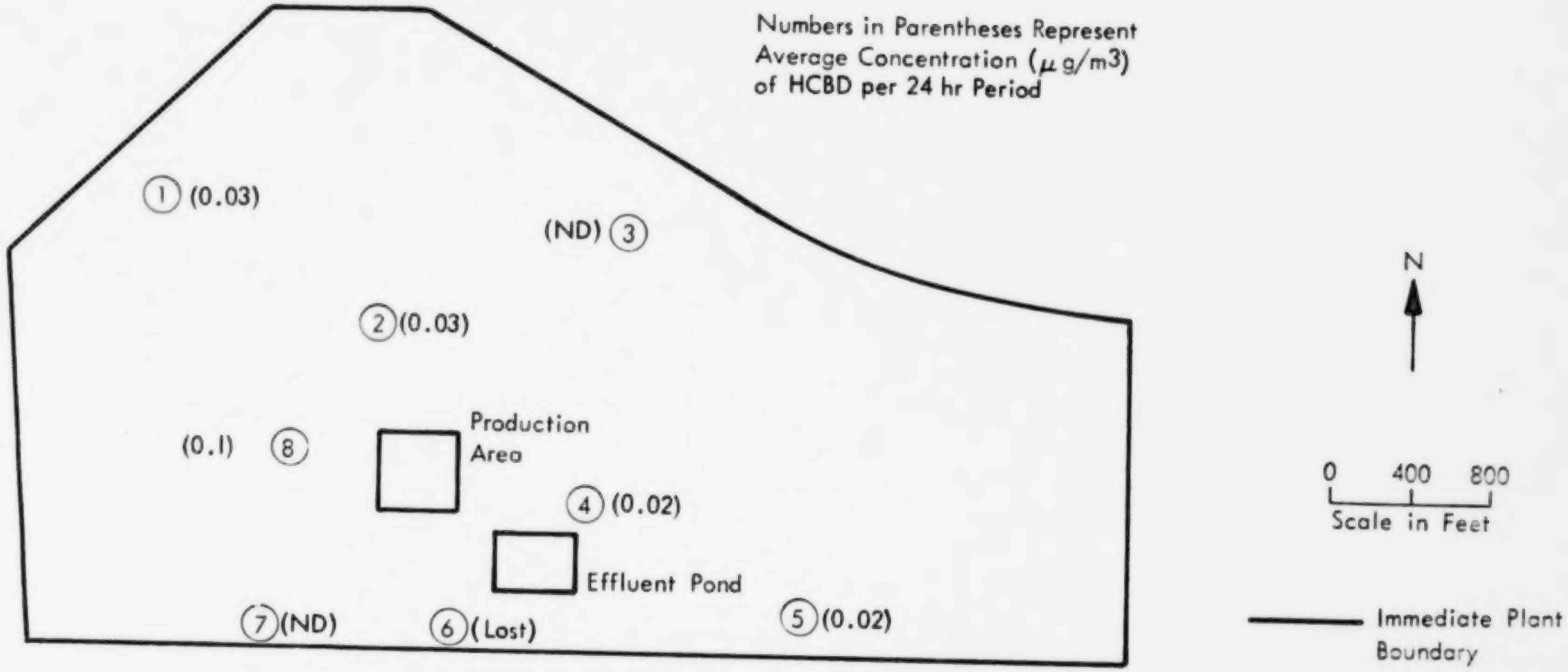

Numbers in Parentheses Represent Average Concentration (μg/m3) of HCBD per 24 hr Period
① (0.03)
(ND) ③
②(0.03)
(0.1) ⑧
Production Area
④ (0.02)
Effluent Pond
⑦(ND)
⑥(Lost)
⑤(0.02)
N
0 400 800
Scale in Feet
Immediate Plant Boundary

Figure 13. Average concentrations of HCBD in air at eight sampling stations at Ciba-Geigy Corporation, St. Gabriel, Louisiana

Soil Samples

Four soil samples were collected from the general areas of Air Sampling Stations 1, 3, 5, and 7, respectively. HCBD was not detected in any of the samples.

Water Samples

Two grab samples were collected, raw plant water and process effluent. HCBD was not detected in either sample.

Plant Summary

Concentrations of HCBD in the plant air were very low--from nondetectable to 0.096 $\mu g/m^3$. No HCBD was detected in the soil. Since HCBD was not detected in the process effluent, the water discharged into the Mississippi River contains less than 5 parts per trillion of HCBD.

OLIN CORPORATION, MCINTOSH, ALABAMA

Field sampling at Olin Corporation's pentachloronitrobenzene (PCNB) plant at McIntosh, Alabama, was conducted on August 18, 1975. This plant was sampled predominately for HCB. The samples were, however, analyzed for HCBD and the results are included in this report.

A total of 24 air, including 8 filters and 16 Tenax®-GC columns, 12 soil and sediment, and 10 water samples was collected. In addition to the PCNB plant, this facility included a chlorine production plant which used graphite electrodes in the production process.

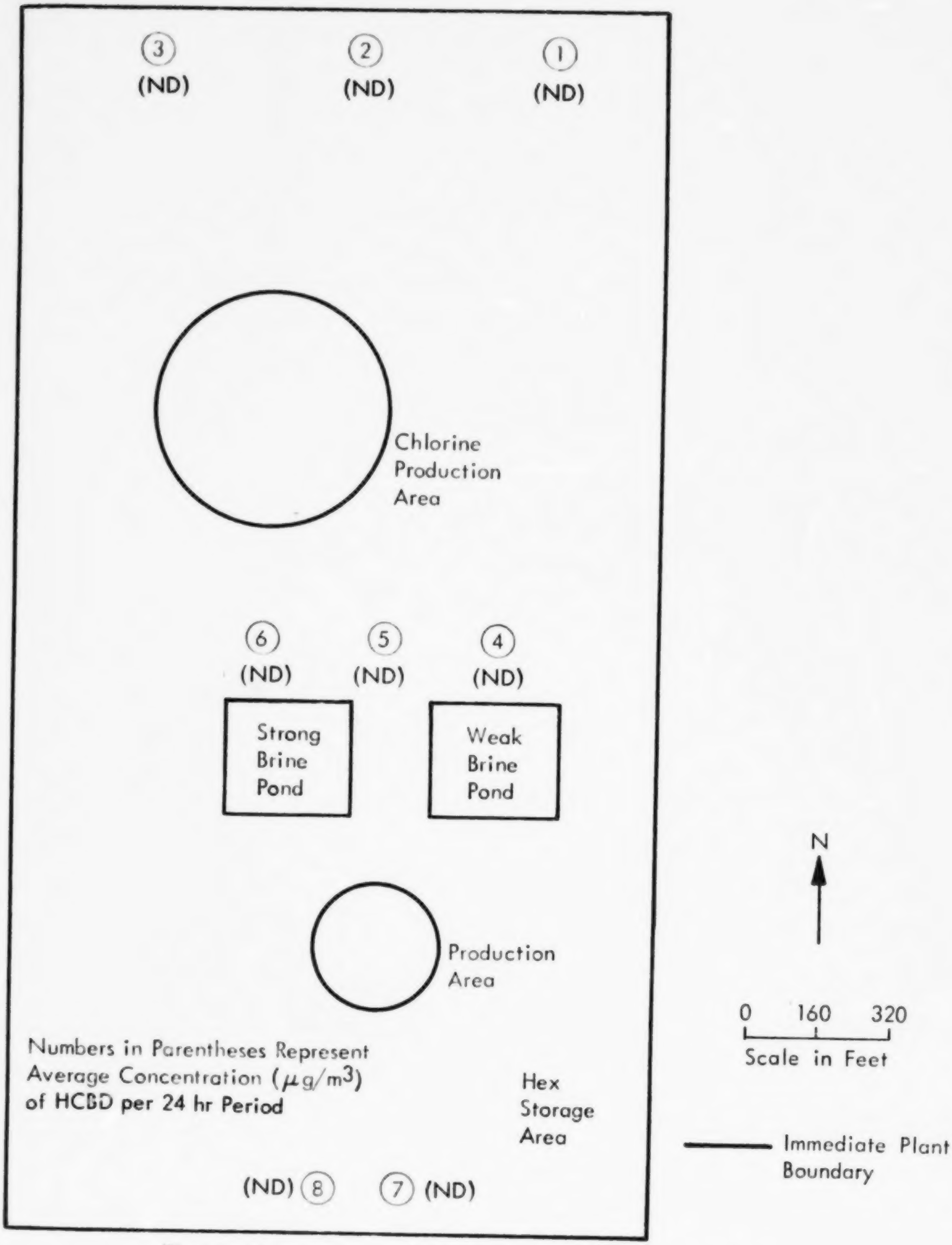
Air Samples

The 24 air samples were collected from eight samplers. Two were positioned at the southern boundary, 3 were in the mid-plant area, north of the PCNB production plant, and 3 were at the northern boundary. Each sampling train consisted of a millipore filter and two Tenax®-GC columns. The samplers were operated 3 hr of each 8-hr period for three 8-hr periods. The wind direction during the sampling was varied so that no upwind-downwind stations could be designated.

Sources and Levels of HCBD - No HCBD was detected in any of the filters or Tenax®-GC traps from the sites shown in Figure 14.

Soil and Sediment Samples

The 12 soil and sediment samples were collected from plant boundaries, transportation routes, landfill and storage areas.


③
(ND)
②
(ND)
①
(ND)
Chlorine Production Area
⑥
(ND)
⑤
(ND)
④
(ND)
Strong Brine Pond
Weak Brine Pond
N
Production Area
0
160
320
Scale in Feet
Hex Storage Area
Immediate Plant Boundary
(ND) ⑧
⑦ (ND)

Numbers in Parentheses Represent Average Concentration ($\mu g/m^3$) of HCBD per 24 hr Period

Figure 14. Average concentrations of HCBD in air at eight sampling stations at Olin Corporation, McIntosh, Alabama

The results of the analysis of these samples are shown in Table 16. HCBD was found in only three samples in a range of 0.006 to 0.009 μg/g. The "old landfill site" was previously used for disposal of "hex" wastes.

Water Samples

Ten water samples were collected from ditches, the nearby creek, the settling pond, the solar pond, and the two brine ponds (strong and weak). No HCBD was detected in any of the samples.

Plant Summary

From the analysis of the air samples, it was determined that this production facility is not a source of HCBD. Only three soil or sediment samples had detectable levels of HCBD while none of the water samples contained HCBD.

PPG INDUSTRIES, INC., LAKE CHARLES, LOUISIANA

Field sampling at PPG Industries' trichloroethylene and perchloroethylene plant at Lake Charles, Louisiana, was conducted on September 4, 1975. A total of 30 air, including 10 filter, 20 Tenax®-GC columns, 7 soil and sediment, and 7 water samples was collected.

Air Samples

The 30 air samples were collected from 10 samplers which circled the plant. The samplers were not positioned in an upwind-downwind array because varied wind direction was expected from information obtained during the presampling site visit. However, the wind was predominately from the east and east-southeast during sampling. Samplers were also positioned to take advantage of existing electrical outlets. The sampling was conducted for an integrated 24-hr period; two Tenax®-GC columns in tandem were used.

Source and Levels of HCBD - The results of the analysis are shown in Table B-7 in Appendix B. A simplified plant map with the sampling locations and the 24-hr average concentrations (Tenax®-GC plus filter) of HCBD is shown in Figure 15. HCBD concentrations were from nondetectable at Stations 7 and 8 to 0.55 $\mu g/m^3$ at Station 10.

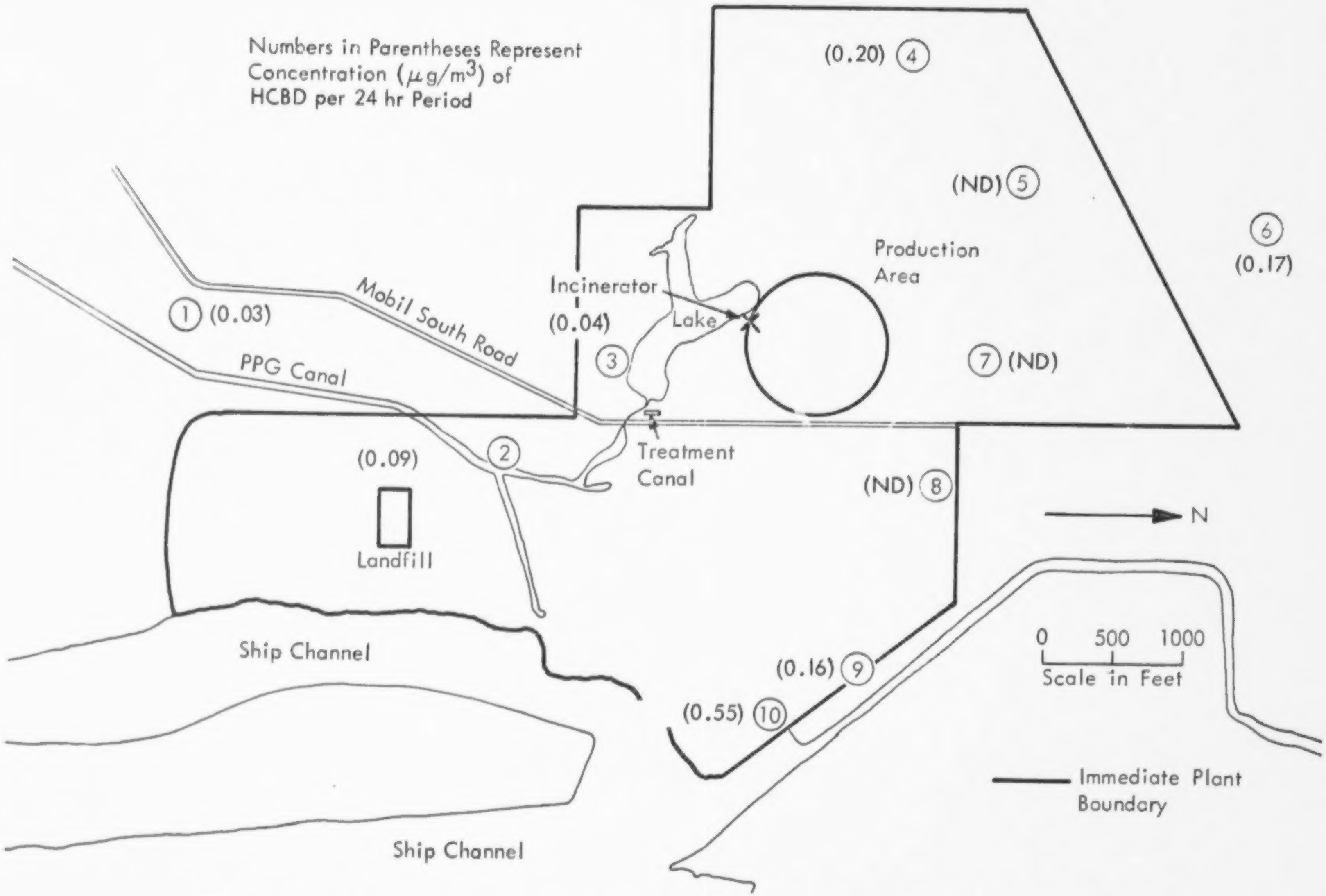
The pattern of HCBD distribution indicates that the primary source of HCBD is not the plant or incinerator. Station 10 which showed the highest HCBD concentration was located near the barges which are sometimes used for temporary storage of the "liquid bottoms" resulting from the production process when repair or modification of the incinerators is required.[4] Detectable levels of HCBD at the stations downwind of the old landfill site and downwind of the incinerator suggests that they are also sources. All HCBD was detected in the vapor form only.

Table 16. HCBD CONCENTRATIONS IN SOIL AND SEDIMENT OLIN CORPORATION, MCINTOSH, ALABAMA

Samples		Concentration (µg/g) HCBD
Soil		
S-1	Northern boundary road	0.006
S-2	Old landfill (northeastern boundary)	0.008
S-3	Brine pond area	ND
S-4	Center road (running north/south)	0.009
S-5	High-lift route (organic plant to storage area)	ND
S-6	Southeast landfill	ND
S-7	"Hex" storage area	ND
S-8[a]	Old "Hex" dump area	
S-9	East road	ND
S-10	West road	ND
S-11	South road	ND
Sediments		
Strong brine pond sediment		ND

---

[a] This sample is mostly tar which is used to cover the general old "Hex" area; the extracted solution was so dirty that analysis was not possible even after cleanup.



Numbers in Parentheses Represent
Concentration ($\mu g/m^3$) of
HCBD per 24 hr Period
(0.20) ④
(ND) ⑤
⑥
(0.17)
Production
Area
Incinerator
(0.04)
Lake
① (0.03)
Mobil South Road
PPG Canal
③
⑦ (ND)
(0.09)
②
Treatment
Canal
(ND) ⑧
N
Landfill
Ship Channel
(0.16) ⑨
(0.55) ⑩
0 500 1000
Scale in Feet
Immediate Plant
Boundary
Ship Channel

Figure 15. Average concentrations of HCBD in air at 10 sampling stations at PPG Industries, Lake Charles, Louisiana

Soil Samples

The four soil samples were collected at the plant boundaries, plant roads, and the landfill. The results of the analysis are shown in Table 17. The levels of HCBD in the soil ranged from 0.008 μg/g around Air Sampling Stations 8 through 10 to 6.4 μg/g at the landfill area.

Analysis of the soils from the Mobil South Road (0.34 μg/g HCBD) and the northern plant boundary along Air Sampling Stations 4, 5, and 7 (0.29 μg/g HCBD) gave similar results.

The elevated levels of HCBD at Stations 4, 5, and 7 are consistent with a prevailing east and east-southeast wind resulting in particulate fallout and vapor condensation from the incinerator and production plant. The concentration detected in the Mobil South Road composite sample indicates the PPG canal is a possible source of HCBD. The results of sediment and water analyses discussed below verify this possibility.

Sediment Samples

The three sediment samples were taken from the PPG canal (near Air Sampling Station 1), the ship channel, and the main effluent from the organic plant. HCBD was detected in all three sediment samples; the lowest level (0.04 μg/g HCBD) was in the ship channel and the highest level (33.1 μg/g HCBD) was at the organic plant effluent. The presence of HCBD in the ship channel sediment may be associated with waste loading into the barges.

The presence of a significant amount of HCBD (0.93 μg/g) in the downstream PPG canal sediment indicates accumulation of deposits from the organic plant effluent. This sediment was collected 1,000 ft beyond Air Sampling Station 1, i.e., at least 4,000 ft from the organic plant effluent.

Water Samples

The seven water samples were collected from the lake (incinerator feed), incinerator scrubber, treatment canal, landfill (standing water), PPG canal, and ship channel.

The results of the analysis of the seven water samples are shown in Table 18. The highest concentrations of HCBD (240 μg/liter) were found in the treatment canal inlet sample. The treatment canal outlet contained HCBD levels at 75 μg/liter, indicating that the treatment removed about 60% of the HCBD. The sample collected downstream of the PPG canal near Air Sampling Station 1 contained HCBD levels at 11 μg/liter. The observation of such a relatively high concentration of HCBD in the canal water at more than 3,000 ft downstream from the effluent point is consistent with the level detected in sediments collected 1,000 ft further downstream.

Table 17. HCBD CONCENTRATIONS IN SOIL AND SEDIMENT FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Samples	Concentration (μg/g) HCBD
Air Stations 4, 5, 7 soil composite	0.29
Air Stations 8, 9, 10 soil composite	0.008
Mobil south road	0.34
Landfill	6.4
PPG sediments	
Sediment 1 (downstream PPG canal)	0.93
Sediment 2 (main organic plant effluent)	33
Ship channel sediment	0.04

Table 18. HCBD CONCENTRATIONS IN WATER FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sample	Concentration (μg/ℓ) HCBD
Incinerator feed water (lake water)	0.08
Scrubber water	0.04
Inlet (treatment canal)	244
Outlet (treatment canal)	75
Surface water (landfill)	125
Downstream PPG cnaal (Mobil Bridge No. 1)	11
Ship channel (next to Air Station No. 10)	0.52

Note: ND = none detected.

The lake water (incinerator feed water) contained 0.08 μg/liter HCBD. The scrubber water from the incinerator contained a lower level of HCBD (0.04 μg/liter) than the feed water and the treatment canal inlet. Therefore, the contribution of the incinerator scrubber water to levels of HCBD in the PPG canal appears to be negligible.

Plant Summary

The primary source of HCBD in air appears to be the "liquid bottom" storage in the ship channel while the incinerator, plant effluent water, and the old landfill are secondary sources. HCBD was detected only as a vapor. The inlet and outlet water to the treatment canal contained 244 and 75 μg/liter of HCBD, respectively. The canal, 3,000 ft downstream from the organic plant effluent, still contained 11 μg/liter HCBD. Sediment samples at the organic plant effluent and 4,000 ft down the canal contained 33 and 0.9 μg/g HCBD, respectively. The PPG canal flows into Lake Charles.

SECTION VI

SUMMARY AND CONCLUSIONS FOR PROGRAM TASK IB

SUMMARY

A summary of results is listed in Table 19. The high and low concentrations of HCBD are listed for each type of sample, along with the products and waste-disposal methods for each site.

Figure 16 shows the highest levels of HCBD in air and the levels detected in the samples taken the greatest distance downwind from the suspected source(s) at each plant. Sampling distances from each source are shown in parentheses.

In general, of the six industries sampled, higher concentrations of HCBD were associated with the production of perchloroethylene and trichloroethylene. Because most of the chlorinated hydrocarbon plants produced a combination (perchloroethylene, trichloroethylene, carbon tetrachloride, etc.) of products, it is difficult to extrapolate the results obtained at a particular plant to a single product. In the one plant that produced only carbon tetrachloride, the HCBD levels were quite low. No. HCBD was detected in samples from the pentachloronitrobenzene production plant. The levels of HCBD associated with plants producing chlorine and triazine herbicides were very low.

Several different waste-disposal methods were used at the perchloro- and trichloroethylene plants that were sampled. They included off-site and on-site landfill combined with open pit or pond storage; and off-site and on-site incineration. The highest level of HCBD was detected in air and soil at the plant using on-site landfill and open pit storage. High HCBD levels were detected in loading and transfer areas at plants using off-site disposal methods. Lower levels of HCBD were found at plants using on-site incineration but downwind air concentrations were still elevated above background at both plants. The lowest levels of HCBD for perchloro- and trichloroethylene production plants were detected at the plant using production by low temperature oxychlorination and the on-site incineration of liquid bottom wastes.

Table 19. DATA SUMMARY FOR PROGRAM TASK NO. 1

Company	Products	Substance	Air ($\mu g/m^3$) High	Air ($\mu g/m^3$) Low	Water ($\mu g/\ell$) High	Water ($\mu g/\ell$) Low	Soil ($\mu g/g$) High	Soil ($\mu g/g$) Low	Sediment ($\mu g/g$) High	Sediment ($\mu g/g$) Low	Waste disposal
Vulcan Materials Company Wichita, Kansas	Perchloroethylene Carbon tetrachloride Chlorine	HCBD	460	0.05	230	ND	980	0.005	No sample		On-site landfill, and deep well
Stauffer Chemical Company Louisville, Kentucky	Perchloroethylene Carbon tetrachloride Methylen chloride Chloroform, chlorine	HCBD	35	0.17	25	0.1	29	0.001			Off-site landfill
Dow Chemical Company Pittsburg, California	Perchloroethylene Carbon tetrachloride Chlorine	HCBD	2.0	ND	No sample		0.005	ND	No sample		Incineration
E. I. du Pont de Nemours Corpus Christi, Texas	Carbon tetrachloride	HCBD	0.03	0.003	0.32	ND	0.004	ND	0.06	ND	On-site landfill and off-site disposal
Diamond Shamrock Deer Park, Texas	Trichloroethylene Perchloroethylene Chlorine	HCBD	2.4	0.09	2.0	ND	19	0.06	No sample		Off-site incineration
Olin Corporation McIntosh, Alabama	Pentachloronitrobenzene Chlorine	HCBD	ND	ND	ND	ND	0.009	ND	ND	Only one sample	Solid wastes (in blocks) stored in open field covered with plastic
Ciba-Geigy Corporation St. Gabriel, Louisiana	Atrazine Propazine Simazine	HCBD	0.1	ND	ND	ND	ND	ND	No sample		Off-site incineration
PPG Industries Lake Charles, Louisiana	Trichloroethylene Perchloroethylene Vinyl chloride Vinylidene chloride Chlorine, etc.	HCBD	0.55	ND	240	0.04	6.4	0.008	33	0.04	Incineration, landfill, and treatment canal
Linden Chlorine Linden, New Jersey	Chlorine	HCBD	No sample		0.08	ND	ND	Only one sample	0.18	0.04	Holding pond

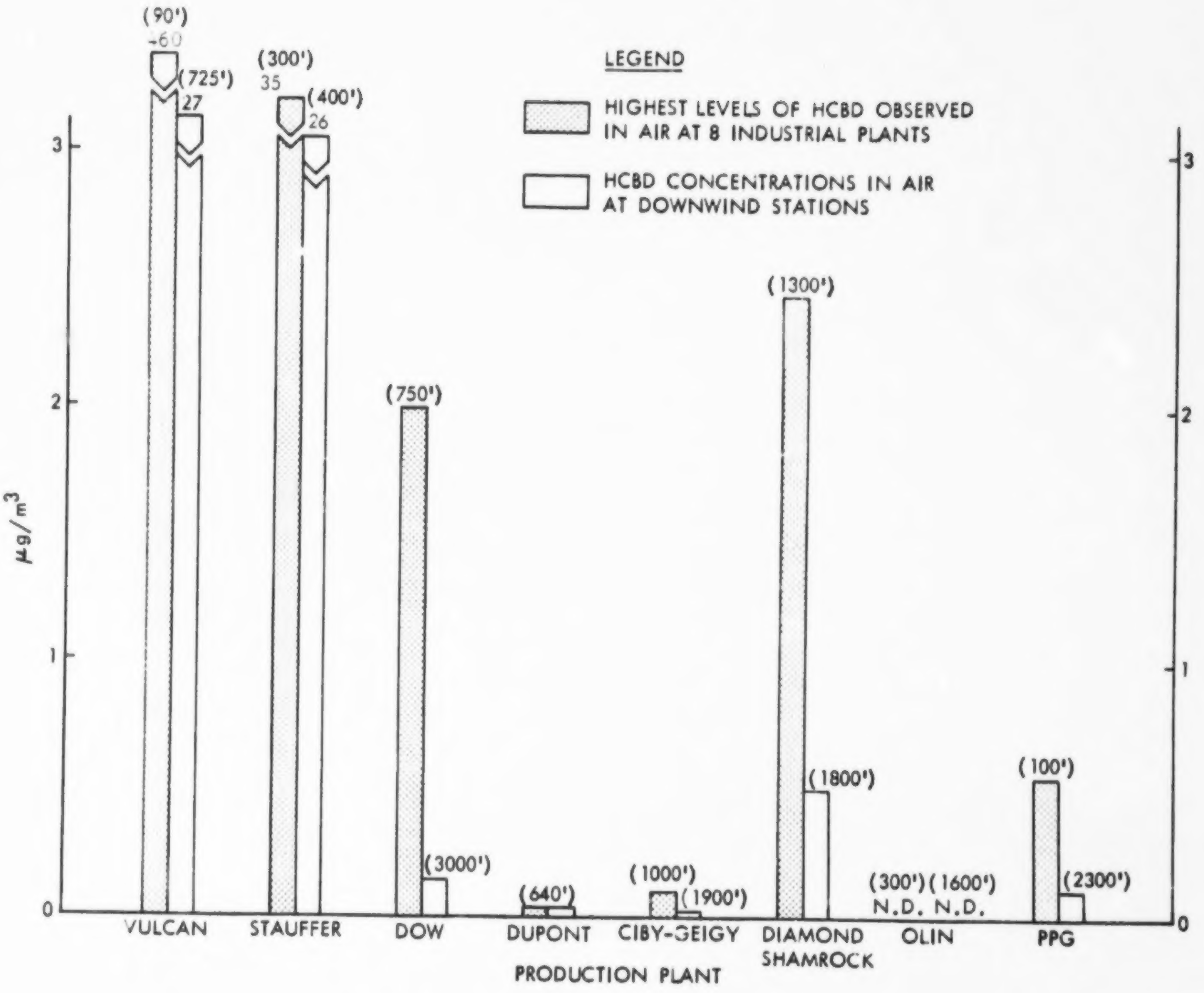


Figure 16. Summary of HCBD concentrations in air

The production of perchloro- and trichloroethylene by low temperature oxychlorination and the incineration of liquid bottom wastes resulted in a unique HCBD ratio in the air and water samples. Generally, high levels of HCBD in the air are associated with high levels in water. Conversely, low levels in the air are accompanied by low levels in water. The low temperature process resulted in low HCBD levels in the air but very high levels in the water. Levels of 10 μg/liter HCBD were found in treated process water more than 3,000 ft from the plant effluent.

The overall relative standard deviation of the air sampling and analysis procedure, i.e., sample collection, storage, extraction, and analysis, was calculated to be less than 20%, based on determinations of HCBD levels from sample pairs positioned at the same distances, but at different heights from the emission source. HCBD was collected efficiently on a single Tenax®-GC plug for air sample volumes up to 2,000 liters; HCBD breakthrough was observed at 4,000 liters. Good agreement was obtained from the analysis of water samples collected by "grab" sampling and by concentration of HCBD on XAD-4 resin.

CONCLUSIONS

Industrial Sources of HCBD

Considering the estimated production volumes of each of the six industries and concentrations detected in this study, perchloroethylene and trichloroethylene production was easily the most significant source of HCBD for the industries sampled. Although the total volume of chlorine produced was estimated to be 20 times that of perchloroethylene and trichloroethylene combined, the production of chlorine did not appear to be a significant source of HCBD. Carbon tetrachloride production alone did not appear to be a significant source of HCBD but this conclusion could be biased by the fact that the single-product carbon tetrachloride plant was the newest facility that was sampled (on-line in 1973). The production of PCNB did not result in the release of detectable levels of HCBD. Estimated triazine herbicide production volumes and the associated HCBD levels determined in this study were very low; therefore, the production of these compounds is not a significant source of HCBD.

Effects of Waste Disposal Methods

In general, methods that involve open storage (pits, lagoons, etc.) resulted in elevated levels of HCBD in air and surrounding soil. Waste holding areas were often the most significant emission source within the plant area. Contaminated soil appeared to be a secondary source of HCBD at two sites. On-site incineration resulted in elevated air--HCBD levels for 750 and 2,300 ft, respectively, at two sites. Elevated HCBD levels at the latter site (~ 0.2 $\mu g/m^3$) extended at least 3,000 ft downwind of the incinerator. The HCBD levels in water were reduced from 0 to 70% at three plants that passed liquid wastes through holding ponds or treatment canals.

REFERENCES

1. Food and Drug Administration, Compliance Program Guidance Manual, Program 7320.30, Project Code 05, Problem Code FH-18, "HCBD in Foods Survey."

2. Mumma, C. E., and E. W. Lawless, "Survey of Industrial Processing Data: Task I - Hexachlorobenzene and Hexachlorobutadiene Pollution from Chlorocarbon Processes," Final Report by Midwest Research Institute on Contract No. 68-01-2105 for the Environmental Protection Agency, June 1975.

3. "Sampling and Analysis of Selected Toxic Substances: Task IA - Hexachlorobenzene," Final Report by Midwest Research Institute on Contract No. 68-01-2646 for the Environmental Protection Agency, June 1976.

4. Personal communication with Mr. C. A. Burns, Environmental Control Specialist, PPG Industries, Lake Charles, Louisiana, November 1975.

APPENDIX A

PRESAMPLING SURVEY AND FIELD SAMPLING

Presampling surveys and field sampling were conducted on the recommended industrial plants according to the schedule shown in Figure A-1. Essentially, during the presampling survey, information such as the surrounding terrain, meteorological conditions, production technology, and waste disposal technique was gathered. Following each site visit, a detailed field sampling strategy was devised and carried out approximately 2 to 4 weeks after the presampling survey date. Presented below are detailed descriptions of the presampling survey and field sampling conducted at each plant.

VULCAN MATERIAL COMPANY, WICHITA, KANSAS

PRESAMPLING SITE SURVEY

The presampling site survey at Vulcan Materials Company's Wichita, Kansas, plant was conducted on May 6, 1975. The following personnel were present:

Mr. J. I. Jordan, Jr.	Manager, Research and Development, Vulcan Materials Company
Mr. R. A. Bondurant, Jr.	Director, Environmental Control Safety, Vulcan Materials Company
Mr. Dave Harrison	Acting Technical Manager, Wichita Plant, Vulcan Materials Company
Mr. P. Constant	Midwest Research Institute
Mr. P. Kuykendall	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Vulcan Materials Company is located approximately 7 miles southwest of downtown Wichita and approximately 4 miles from any major residential area. The surrounding terrain is level with only one nearby water source, Cowskin Creek. The prevailing wind in May is generally from the south, southeast or southwest.

Perchloroethylene is produced by the reaction of hydrocarbons and chlorine. The hydrocarbons are generally of a widely variable composition and are obtained from many sources. The chlorine is produced by Vulcan and piped directly from their liquification station to the perchloroethylene reaction pot. Their chlorine production utilizes diaphragm cells and approximately 25% of their anodes are graphite, the remainder being dimensionally stabilized anodes.

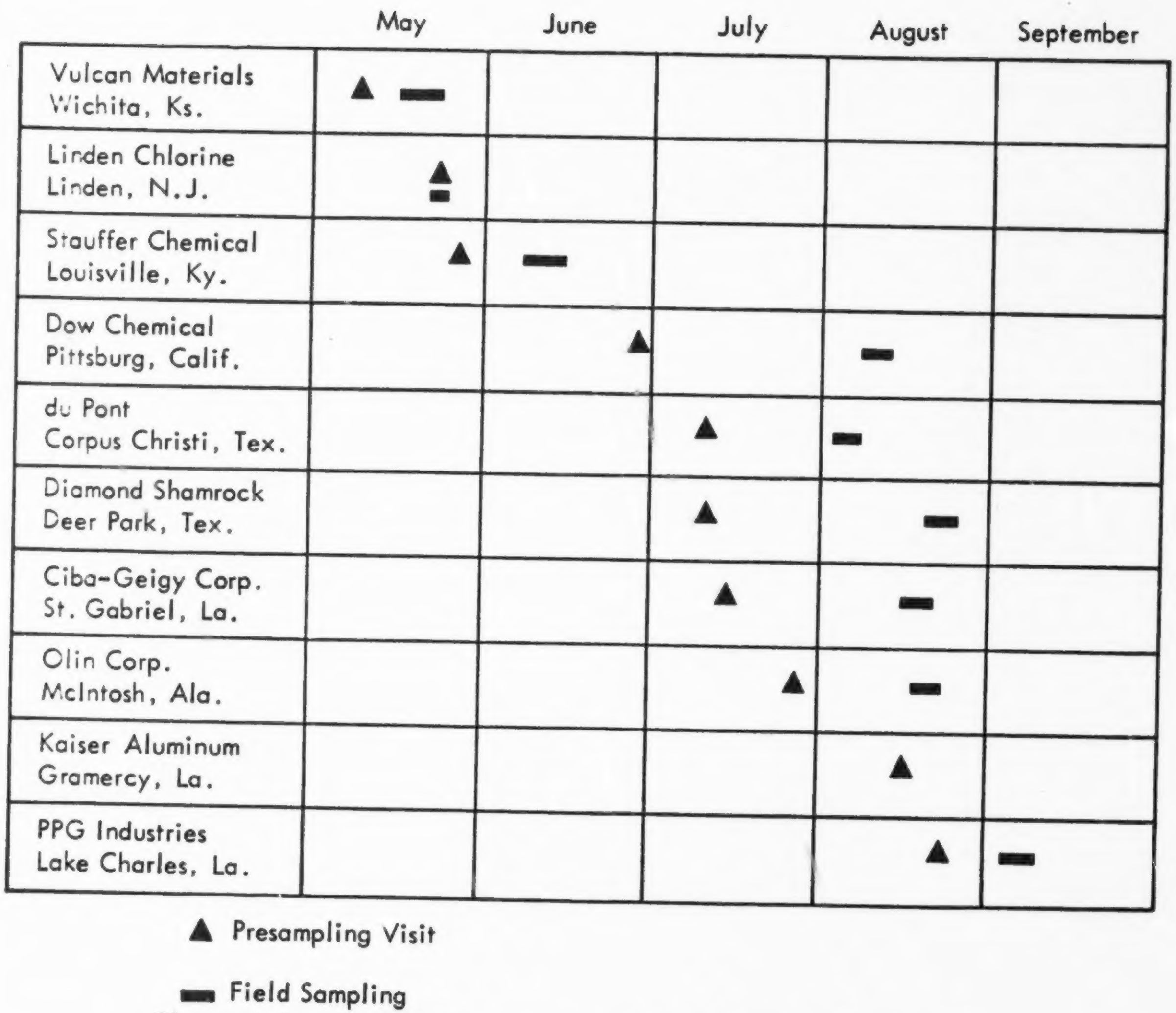


Figure A-1. Presampling survey and field sampling schedule

Vulcan uses two types of waste disposal, deep wells and landfills. The deep wells are used for the disposal of storm runoff, while the landfills are used for the disposal of "heavy ends" waste from perchloroethylene production. The residues are collected in a sealed vessel, transferred to another sealed vessel mounted on a trailer, and transported to an open pit where they are stored under water. When the residue level in the pit reaches a certain level, it is transferred by means of a backhoe to a dump truck and transported to the landfill, which is located southeast of the plant. There it is dumped, covered with polyethylene sheeting, then covered with dirt.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of May 19, 1975.

FIELD SAMPLING

Field sampling at the Vulcan plant was conducted on May 20, 1975. Air, soil, and water samples were collected as planned. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Five general areas were chosen for air sampling: (a) perchloroethylene plant; (b) "Hex Pit;" (c) solar pond - landfill; (d) downwind of these locations; and (e) upwind of these locations. The total sampling time was divided into 4-hr periods. The upwind and downwind samplers were operated continuously during each 4-hr period, whereas all other samplers were operated only for the 1st hr of each 4-hr period. Each sampling location is shown in Figure A-2. Exact location was measured with respect to the suspected emission source and was reported along with other sampling data in Table A-1.

Soil Sampling

Soil sampling covered eight general areas:

S-1	Around the perchloroethylene plant
S-2	Route from perchloroethylene plant to "Hex Pit"
S-3	Around the "Hex Pit"
S-4	Route from "Hex Pit" to landfill
S-5	Between old and current landfill sites

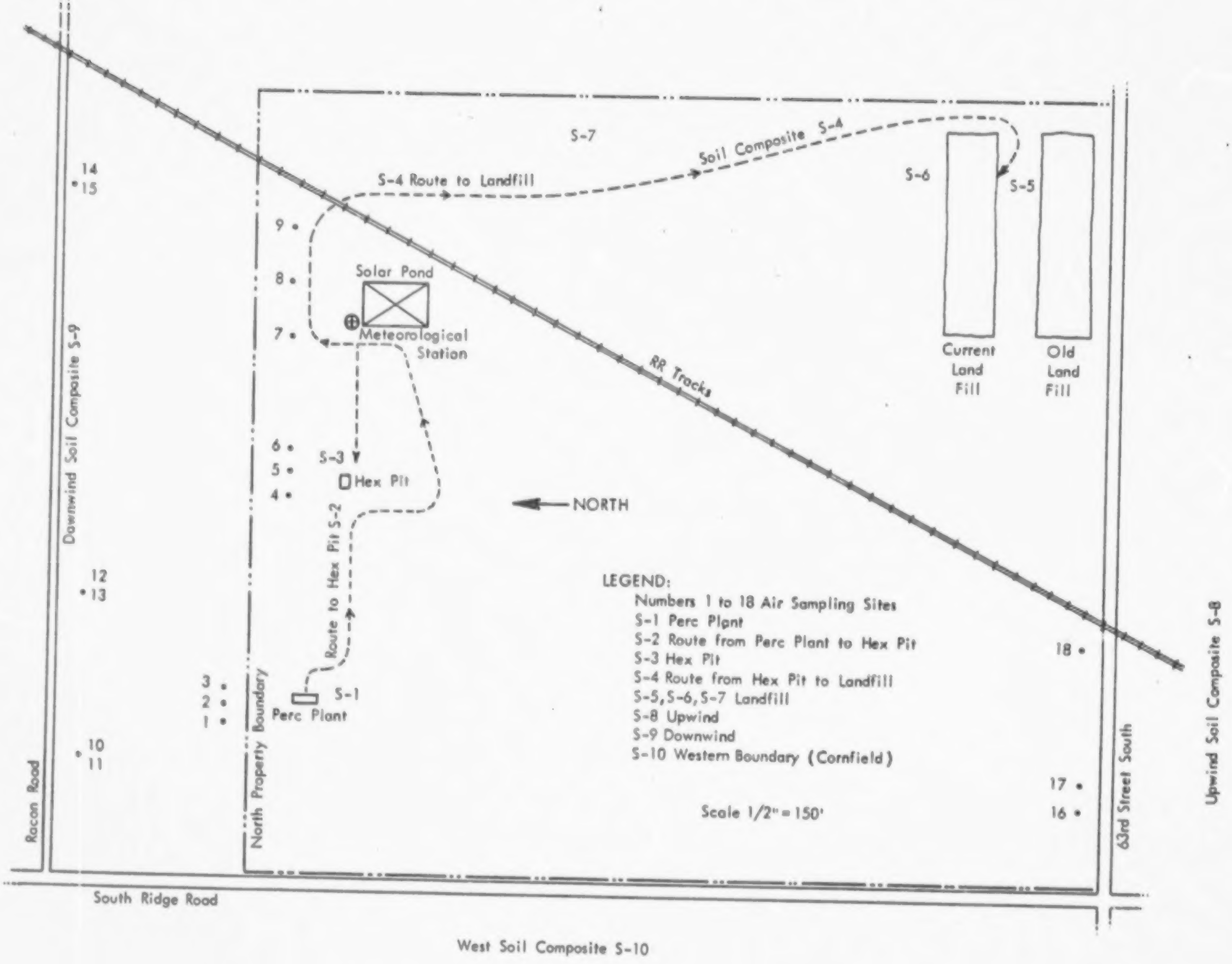

Downwind Soil Composite S-9
Racon Road
South Ridge Road
West Soil Composite S-10
63rd Street South
Upwind Soil Composite S-8
North Property Boundary
RR Tracks
S-7
Soil Composite S-4
S-4 Route to Landfill
S-6
S-5
Current Land Fill
Old Land Fill
Solar Pond
Meteorological Station
S-3
Hex Pit
Route to Hex Pit S-2
S-1
Perc Plant
NORTH
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
LEGEND:
Numbers 1 to 18 Air Sampling Sites
S-1 Perc Plant
S-2 Route from Perc Plant to Hex Pit
S-3 Hex Pit
S-4 Route from Hex Pit to Landfill
S-5, S-6, S-7 Landfill
S-8 Upwind
S-9 Downwind
S-10 Western Boundary (Cornfield)
Scale 1/2" = 150'

Figure A-2. Sampling locations at Vulcan Materials Company - Wichita plant

Table A-1. AIR SAMPLING DATA AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

General area	Sample no.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Total sample vol. (ℓ)	Sampler height (ft)
"Perc Plant"	1	250 ft north of "Perc Plant"- 250 ft west of Sample No. 2	1st hr of 4 hr	19.5	0.5	178	11
	2	250 ft north of "Perc Plant"	1st hr of 4 hr	19.5	0.5	149	11
	3	250 ft north of "Perc Plant"- 50 ft east of Sample No. 2	1st hr of 4 hr	19.5	0.5	207	11
"Hex Pit"	4	150 ft north of "Hex Pit" 75 ft west of Sample No. 2	1st hr of 4 hr	19.5	0.5	156	4
	5	150 ft north of "Hex Pit"	1st hr of 4 hr	19.5	0.5	232	4
	6	150 ft north of "Hex pit"- 75 ft east of Sample No. 2	1st hr of 4 hr	19.5	0.5	195	4
Solar pond-landfill	7	1,500 ft north of landfill- 225 ft northwest of solar pond	1st hr of 4 hr	19.5	3.5	813	4
	8	1,500 ft north of landfill- 225 ft north of solar pond	1st hr of 4 hr	19.5	3.5	1,123	4
	9	1,500 ft north of landfill- 440 ft northeast of solar pond	1st hr of 4 hr	19.5	3.5	1,198	4
Downwind	10 and 11	525 ft north of plant boundary- 340 ft east of Ridge Road	4 hr	19.5	3.5	3,646	10-4
			4 hr	19.5	3.5	3,862	11-11
	12 and 13	525 ft north of plant boundary- 850 ft east of Ridge Road	4 hr	19.5	3.5	3,930	12-4
			4 hr	19.5	3.5	4,172	13-11
	14 and 15	525 ft north of plant boundary- 2,100 ft north of Ridge Road	4 hr	19.5	3.5	4,291	14-4
			4 hr	19.5	3.5	4,272	15-11
	16	On southern plant boundary- 225 ft east of Ridge Road	4 hr	19.5	3.5	3,744	4
	17	On southern plant boundary- 300 ft east of Ridge Road	4 hr	19.5	3.5	3,176	4
	18	On southern plant boundary- 700 ft east of Ridge Road	4 hr	19.5	3.5	3,353	4

S-6	North of current landfill site
S-7	Along the eastern plant boundary
S-8	Along the southern plant boundary, passed 63rd Street
S-9	Beyond the northern plant boundary, along Racon Road
S-10	Along the western plant boundary, along the cornfield

All the samples were composites except the landfill samples. In addition, a sample of the "Hex Pit" solids was also collected.

Water Sampling

Water samples were taken from four general areas:

1. Upstream from waste inflow - Cowskin Creek
2. Downstream from waste inflow - Cowskin creek
3. Solar pond water
4. "Hex Pit" water

The location of the solar pond and "hex" pit is shown in Figure A-2; the sampling locations in Cowskin Creek are shown in Figure A-3.

Samples from Cowskin Creek were collected on Amberlite XAD-4 resin via a battery-operated pump. Grab samples were taken from the solar pond and "Hex Pit."

Plant Activities and Weather Conditions

Plant activities were observed during the sampling period and are tabulated in Table A-2.

The weather conditions during the sampling period are summarized in Table A-3. Fortunately, the wind was from the south or southeast during the entire sampling period except for the last 4-hr interval. The change in wind direction coincided with a thunderstorm which forced a termination of sampling during the final 4-hr period.

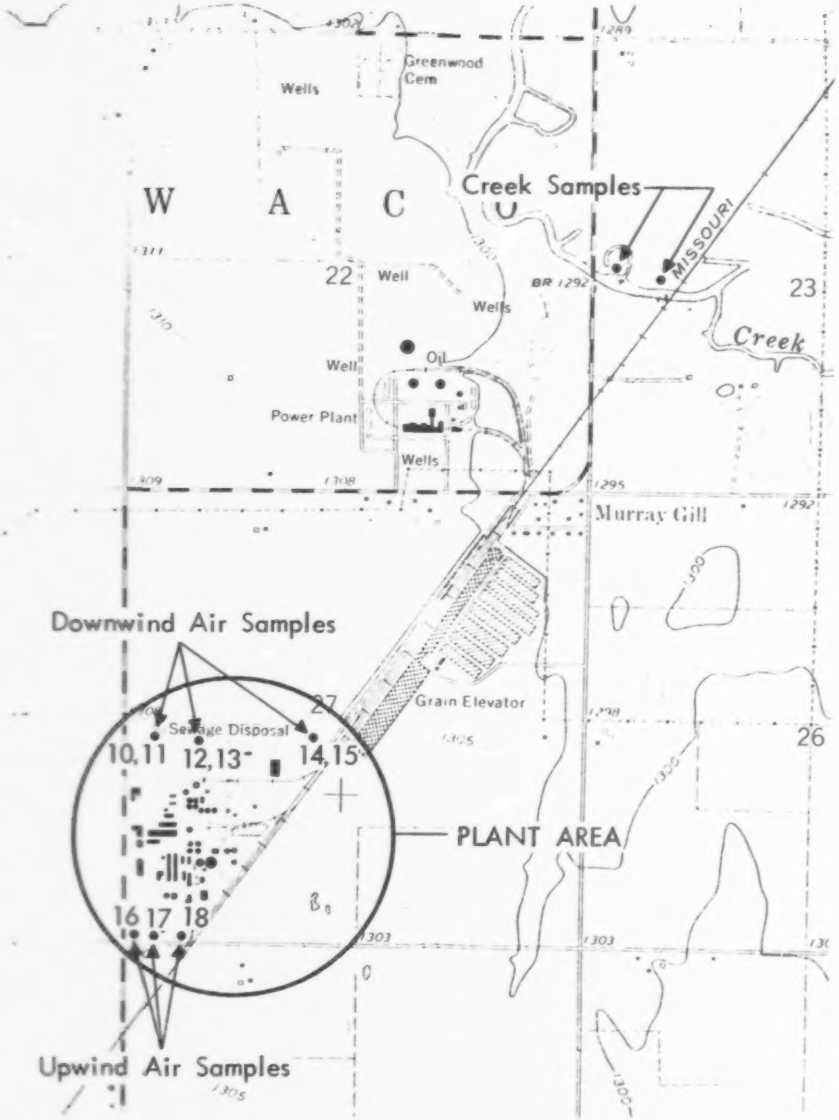

Greenwood Cem
Wells
WACO
Creek Samples
MISSOURI
22
Well
Wells
BR 1292
23
Creek
Well
Oil
Power Plant
Wells
1308
1295
Murray Gill
1292
Downwind Air Samples
27
Grain Elevator
Sewage Disposal
10,11
12,13
14,15
26
PLANT AREA
16 17 18
1303
1303
Upwind Air Samples
1305

Figure A-3. Vulcan Materials Company - Wichita plant, and surrounding areas (2.6 in. = 1 mile)

Table A-2. PLANT ACTIVITIES DURING SAMPLING AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Time	"Perc plant"	"Hex pit"	Solar pond landfill	Downwind	Upwind
May 21					
1900	Normal	Fuller than usual	Normal	Normal	Normal
2000	Normal	Fuller than usual	Normal	Normal	Normal
2100	Normal	Fuller than usual	Normal	Normal	Normal
2200	Normal	Fuller than usual	Normal	Normal	Normal
2300	Normal	Fuller than usual	Normal	Normal	Normal
2400	Dumped "Hex"	Fuller than usual	Normal	Dumped "Hex"	Normal
May 22					
0100	Normal	Dumped "Hex"	Normal	Dumped "Hex"	Normal
0200	Normal	Normal	Normal	Normal	Normal
0300	Normal	Normal	Normal	Normal	Normal
0400	Normal	Normal	Normal	Normal	Normal
0500	Normal	Normal	Normal	Normal	Normal
0600	Normal	Normal	Normal	Normal	Normal
0700	Normal	Normal	Normal	Normal	Normal
0800	Normal	Normal	Normal	Normal	Normal
0900	Normal	Normal	Normal	Normal	Normal
1000	Normal	Normal	Normal	Normal	Normal
1100	Normal	Normal	Normal	Normal	Normal
1200	Normal	Normal	Normal	Normal	Normal
1300	Dumped "Hex"	Normal	Normal	Dumped "Hex"	Normal
1400	Normal	Dumped "Hex"	Normal	Dumped "Hex"	Normal
1500	Rain	Rain	Rain	Rain	Rain

Table A-3. WEATHER CONDITIONS DURING SAMPLING AT VULCAN MATERIALS COMPANY, WICHITA, KANSAS

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind Speed	Wind Direction
May 21					
1900	27	722	None	15	South southeast
2000	27	722	None	12	South southeast
2100	24	723	None	11	Southeast
2200	23	724	None	12	Southeast
2300	23	724	None	13	South southeast
2400	23	724	None	12	South
May 22					
0100	23	724	None	11	South southeast
0200	23	724	None	12	Southeast
0300	23	724	None	13	South southeast
0400	23	724	None	10	South southeast
0500	21	723	None	9	South
0600	21	723	None	9	South southeast
0700	23	723	None	9	South southeast
0800	25	725	None	9	South southeast
0900	25	725	None	11	South southeast
1000	26	725	None	10	South
1100	26	725	None	12	South
1200	27	725	None	15	South
1300	29	725	None	15	South
1400	29	725	None	12	South
1500	29	725	Rain	10	Northwest

LINDEN CHLORINE COMPANY, LINDEN, NEW JERSEY

Presampling site survey at Linden Chlorine Company was conducted on May 29, 1975. The following personnel were present:

Mr. Ronald Burkett	Linden Chlorine Plant
Mr. Bill Heineman	Linden Chlorine Plant
Mr. Edward J. Finfer	Environmental Protection Agency, Region II
Mr. Martin L. Sanvito	DEP, BAPC
Mr. William J. O'Sullivan	DEP, Springfield office
Mr. William A. McGough	Central Jersey Regional Air Pollution Control Agency
Mr. Richard Hills	Central Jersey Regional Air Pollution Control Agency
Dr. George Scheil	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Linden Chlorine Company is located in the middle of a heavy industrial area. The closest residential areas are greater than 1 mile away, and nearby water source is the Arther Kill River.

During the visit, it was learned that graphite electrodes are no longer used in production of chlorine, instead platinum-coated titanium electrodes (dimensionally stabilized anodes) have been used since the end of March 1975. Because the potential for producing HCB and HCBD is much less for this type of electrode, it was decided that air sampling was not necessary. However, the holding pond that contains brine sludge and eroded graphite from previously used electrodes may contain significant quantities of chlorinated organics. In addition to carbon from eroded electrodes, the pond also contains spent carbon filters that were used to remove organics from process and surface water which flows through an open ditch to the Arthur Kill River. By far the largest portion of this water is made up of wastes from the nearby Gaf dye plant. The Gaf flume appeared to have a high organic content before the wastes from the chlorine plant enter the stream. Because of possible past HCB and HCBD contamination in the holding pond, samples were taken from the pond and wastewater stream. The samples are described below:

Sample type	Sample location
Water	Holding pond, inlet
Water	Holding pond, outlet
Water	Gaf weir, upstream of Cl_2 plant
Water	Waste stream, downstream of Cl_2 plant
Water	Tap water (control)
Solids	From holding pond, settled and suspended
Solids	Dredged solids adjacent to holding pond
Sludge	Waste stream, downstream of Cl_2 plant

The following samples were sent to Midwest Research Institute, on a later date by the Linden Chlorine Plant: process water, circulating brine, and uncontaminated soil.

STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

PRESAMPLING SITE SURVEY

The presampling site survey at Stauffer Chemical Company's Louisville, Kentucky, plant was conducted on May 30, 1975. The following personnel were present:

Mr. Arthur Wood	Manufacturing Manager, Stauffer Chemical Company
Mr. Harry Kutz	Plant Manager, Louisville plant
Mr. Kenneth G. Hebel	EPA/OSHA Testing Coordinator, Eastern Research Center, Dobbs Ferry, New York
Mr. Arthur E. Dungan	Assistant Plant Manager, Louisville plant
Mr. John R. Blunk	Process Superintendent, Louisville plant
Dr. George Scheil	Midwest Research Institute
Dr. J. Spigarelli	Midwest Research Institute

Stauffer Chemical Company is located on the east bank of the Ohio River approximately 6 miles southwest of downtown Louisville, Residential areas surround the plant, the closest being about 1 mile northeast of the plant. The wind direction, according to the weather bureau records (10-year average) at the Louisville airport indicated that during the month of June there is a 50% probability of a south wind (from one of the four southern quadrants) and a much lower probability from any other direction.

The "hex" solids from the perchloroethylene production plant are gravity fed into drums (batch-wise). After several drums (unsealed, possibly covered with a pleastic sheet) accumulate, they are removed from the plant area. Company officials said that the drums are usually removed once a day at 8:00 a.m. The plant operated 24 hr a day, 7 days a week.

The drums are transported to an area just west of the surplus storage building where they are loaded onto trucks and taken to an approved landfill site approximately 15 miles from the plant. Cooling water and surface runoff from the plant area are fed to a sump where the pH is adjusted to 6 to 9, the liquid is pumped to a concrete settling pond, and gravity fed through a pipe into the Ohio River.

Based on the plant operation described above, three possible sources of HCB and HCBD contamination were considered, namely: (a) the production area, especially at the location of the open barrels, (b) the settling pond and (c) the "hex" loading area and the transportation route to the landfill.

FIELD SAMPLING

Field sampling at the Stauffer plant was conducted on June 12, 1975. Air, water, soil, and sediment samples in and around the plant were collected. Detailed descriptions of the sampling and plant activities during sampling are discussed below.

Air Sampling

Eight sampling stations encircling the immediate plant area, and one downwind station were set up. The total sampling period was divided into six 4-hr periods and samplers were operated 2 hr of each 4-hr period. Each sampling location is shown in Figure A-4. Exact locations with respect to the perchloroethylene plant area, are listed along with other sampling data in Table A-4.

Soil Sampling

Soil sampling was conducted in five general areas:

S-1	Upwind (along the southern plant boundary)
S-2	Plant road (along the main road)
S-3	Drum loading area

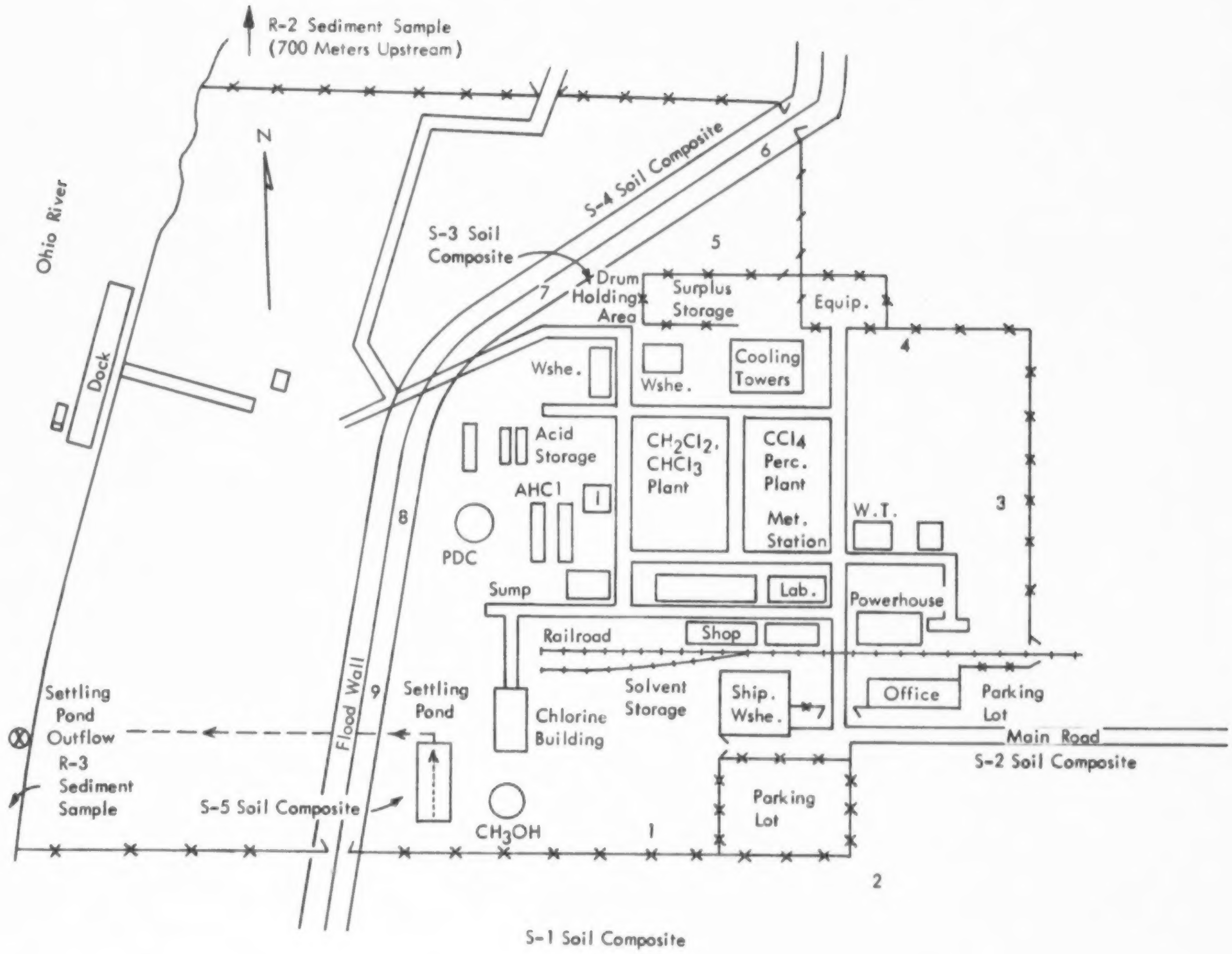

R-2 Sediment Sample
(700 Meters Upstream)
Ohio River
N
Dock
S-4 Soil Composite
6
5
S-3 Soil Composite
7
Drum Holding Area
Surplus Storage
Equip.
4
Wshe.
Wshe.
Cooling Towers
Acid Storage
CH_2Cl_2, $CHCl_3$ Plant
CCl_4 Perc. Plant
Met. Station
AHCl
W.T.
3
8
PDC
Sump
Lab.
Powerhouse
Railroad
Shop
Settling Pond Outflow
R-3 Sediment Sample
Flood Wall
9
Settling Pond
Chlorine Building
Solvent Storage
Ship. Wshe.
7
Office
Parking Lot
Main Road
S-2 Soil Composite
S-5 Soil Composite
CH_3OH
1
Parking Lot
2
S-1 Soil Composite

Figure A-4. Sampling locations at Stauffer Chemical Company - Louisville, Kentucky

Table A-4. AIR SAMPLING DATA AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Total Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	450 ft south southwest of "Perc Plant" area	1st 2 hr of 4 hr	12.3	3.5	2,588	4
Upwind	2	500 ft south southeast of "Perc Plant" area	1st 2 hr of 4 hr	11.8	3.5	2,626	4
East of "Perc Plant"	3	340 ft east of "Perc Plant" area	1st 2 hr of 4 hr	10.6	3.5	2,221	4
Northeast of "Perc Plant"	4	250 ft northeast of "Perc Plant" area	1st 2 hr of 4 hr	12.5	3.5	2,768	4
Downwind	5	300 ft north of "Perc Plant" area	1st 2 hr of 4 hr	12.9	3.5	2,787	4
Downwind	6	400 ft north of "Perc Plant" area	1st 2 hr of 4 hr	13.1	3.5	2,662	4
Northwest of "Perc Plant"	7	330 ft northwest of "Perc Plant" area	1st 2 hr of 4 hr	13.2	3.5	2,850	4
West of "Perc Plant" north of settling pond	8	420 ft west of "Perc Plant" area	1st 2 hr of 4 hr	13.3	3.5	2,872	4
Southwest of "Perc Plant," northwest of settling pond	9	540 ft southwest of "Perc Plant" area	1st 2 hr of 4 hr	13.5	3.5	2.926	4

S-4	Downwind (along the northern plant boundary)
S-5	Settling pond area

All samples were composites.

Water Sampling

Water sampling was limited to the plant well water and the settling pond.

W-1	Plant well water
W-2	Settling pond inlet (24 hr composite)
W-3	Settling pond inlet (grab sample)
W-4	Settling pond outlet (24 hr composite)
W-5	Settling pond outlet (grab sample)
W-6	Settling pond outlet (through Amberlite XAD-4)

Sediment Sampling

Sediment sampling was conducted at three locations:

R-1	Settling pond sediment
R-2	Ohio River, 700 m upstream of Stauffer outflow
R-3	Ohio River, 250 m downstream of Stauffer outflow

Plant Activities and Weather Conditions

Plant activities were observed during the sampling period and are shown in Table A-5.

The weather conditions during the sampling period are summarized in Table A-6.

Table A-5. PLANT ACTIVITIES DURING SAMPLING AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Time	"Perc plant"[a]	Drum loading area	Settling pond
12 June			
1000	Normal	Normal	Normal flow
1100	Normal	Normal	Normal flow
1200	Normal	Normal	Normal flow
1300	Normal	Normal	Normal flow
1400	Normal	"Hex" drums removed	Normal flow
1500	Normal	"Hex" drums removed	Normal flow
1600	Normal	Normal	Normal flow
1700	Normal	Normal	Normal flow
1800	Normal	Normal	Normal flow
1900	Normal	Normal	Normal flow
2000	Normal	Normal	Normal flow
2100	Normal	Normal	Normal flow
2200	Normal	Normal	Normal flow
2300	Normal	Normal	Normal flow
2400	Normal	Normal	Normal flow
13 June			
0100	Normal	Normal	Normal flow
0200	Normal	Normal	Normal flow
0300	Normal	Normal	Normal flow
0400	Normal	Normal	Normal flow
0500	Normal	Normal	Normal flow
0600	Normal	Normal	Normal flow
0700	Normal	Normal	Normal flow
0800	Normal	Normal	Normal flow
0900	Normal	Normal	Normal flow
1000	Normal	Normal	Normal flow

---

[a] Normal operation utilizing HCBD recovery.

Table A-6. WEATHER CONDITIONS DURING SAMPLING AT STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind	
				Speed	Direction
12 June					
1000	26	757	None	4	S
1100	28	757	None	4	S
1200	29	757	None	4	S
1300	29	757	None	6	S
1400	29	757	None	8	S
1500	29	757	None	7	S
1600	29	758	None	6	S
1700	27	758	None	7	S
1800	25	758	None	6	S
1900	24	758	None	6	S
2000	25	759	None	5	S
2100	23	759	None	2	S
2200	21	759	None	5	S
2300	20	759	10 min rain	5	E
2400	20	759	None	4	S
13 June					
0100	19	760	None	4	S
0200	18	760	None	2	S
0300	17	760	None	2	S
0400	16	760	None	0	S
0500	16	760	None	0	S
0600	17	760	None	0	S
0700	17	760	None	0	S
0800	19	760	None	0	S
0900	22	760	None	0	S
1000	25	760	None	0	S

DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

PRESAMPLING SITE SURVEY

Presampling site survey at Dow Chemical Company's Pittsubrg, California, plant was conducted on June 30, 1975. The following personnel were present:

Mr. Ed Elkins	Manager, Environmental and operational Services, Dow Chemical Company
Mr. David Baur	Dow Chemical Company
Mr. Mike Thomas	Dow Chemical Company
Mr. Paul Constant	Midwest Research Institute
Mr. Jim Spigarelli	Midwest Research Institute

Dow Chemical Company is located approximately 2 miles northeast of Pittsburg, California, and 4 miles west of Antioch, California, and is situated on the southern bank of the New York slough of the San Joaquin River. The immediate vicinity is flat, but mountains lie approximately 5 miles south of the plant. Approximately 70% of the time during July the wind is from west or west-northwest. Residential areas are in Pittsburg and Antioch.

Chemicals produced at this Dow facility include chlorine, carbon tetrachloride, and perchloroethylene. According to Mr. Elkins, all wastes from the chlorinated hydrocarbon production flow to a thermal oxidizer, are converted to hydrochloric acid and are recycled. In most instances, surface runoff from the plant areas flow into a solar pond. However, inspection on the plant and the solar pond area were not allowed. Only the plant perimeter and beyond were surveyed.

Air sampling locations were planned based on the thermal oxidizer parameters such as stack gas temperature, gas flow rate, and stack height and diameter.

FIELD SAMPLING

Field sampling at the Dow plant was conducted on August 7, 1975, after several postponements. Air, soil, and water samples were collected. Detailed descriptions of the sampling, plant activities, and weather conditions are discussed below.

Air Sampling

Eight sampling stations were located so that two upwind, three near downwind, and three far downwind samples were obtained. Air was sampled over a 24-hr period with the exception of Stations 2 (upwind) and 7 (far downwind). Two air sampling tubes were operated in series at each station. Each sampling location is shown in Figure A-5. The exact location was measured with respect to the production plant area and is listed along with other sampling data in Table A-7.

Soil Sampling

Soil sampling was conducted in three general areas:

S-1	Upwind (western plant boundary)
S-2	Southern property boundary
S-3	Downwind (eastern plant boundary)

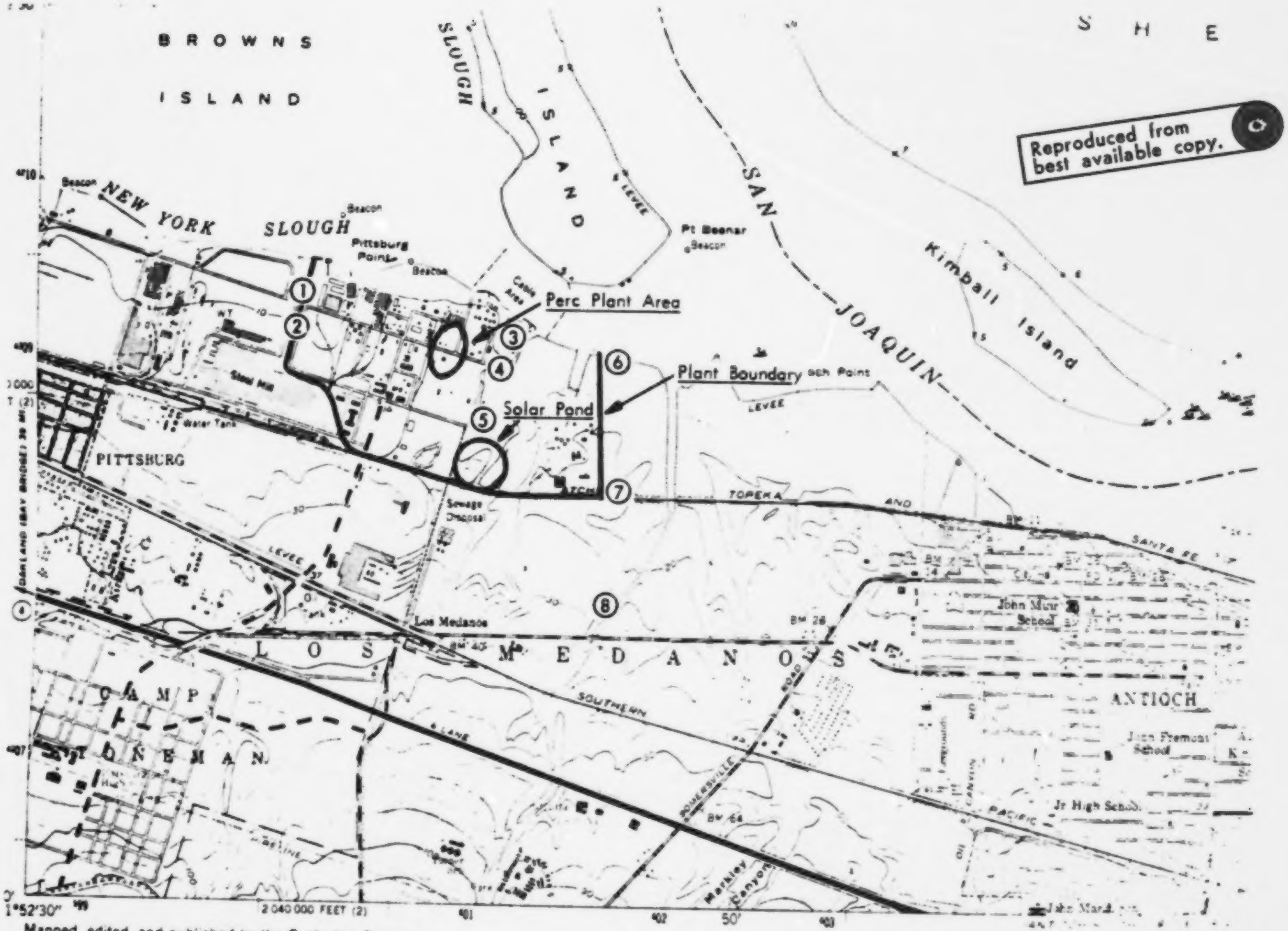
All samples were composites.

Water Sampling

One grab water sample of the New York Slough was obtained at the northeast corner of Dow's property. Water sampling at the solar pond was not permitted by the Dow officials.

Plant Activities and Weather Conditions

Plant activities and weather conditions during sampling are shown in Table A-8.


BROWNS ISLAND
SLOUGH
ISLAND
SAN JOAQUIN
Kimball Island
NEW YORK SLOUGH
Pittsburg Point
Beacon
Pt Beenar Beacon
LEVEE
Cable Area
Perc Plant Area
Plant Boundary
Solar Pond
Steel Mill
Water Tank
PITTSBURG
Sewage Disposal
TOPEKA AND SANTA FE
LEVEE
Los Medanos
LOS MEDANOS
CAMP STONEMAN
SOUTHERN PACIFIC
SOMERSVILLE ROAD
Markley Canyon
John Muir School
ANTIOCH
John Fremont School
Jr High School
①
②
③
④
⑤
⑥
⑦
⑧
2 040 000 FEET (2)
1°52'30"
50'

Reproduced from best available copy.

Mapped, edited, and published by the Geological Survey

Control by USGS, USC&GS, and USCE

Culture and drainage in part by USC&GS from aerial photographs taken

Figure A-5. Sample locations at Dow Chemical Company, Pittsburg, California

Table A-7. AIR SAMPLING DATA AT DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	2,630 ft west-northwest of "Perc Plant"	24 hr continuous	20.7	3.5	4,336	4
Upwind	2	2,780 ft west of "Perc Plant"	24 hr continuous	2.0[a]	3.5	427	4
Near downwind	3	900 ft east-northeast of "Perc Plant"	24 hr continuous	19.8	3.5	4,166	4
Near downwind	4	830 ft southeast of "Perc Plant"	24 hr continuous	18.4	3.5	3,870	4
Near downwind	5	1,280 ft south-southeast of "Perc Plant"	24 hr continuous	17.7	3.5	3,713	4
Far downwind	6	2,550 ft east of "Perc Plant"	24 hr continuous	20.5	3.5	4,314	4
Far downwind	7	3,600 ft southeast of "Perc Plant"	24 hr continuous	4.6[a]	3.5	962	4
Far downwind	8	5,100 ft south-southeast of "Perc Plant"	24 hr continuous	18.9	3.5	3.963	4

---

[a] Generator failure.

Table A-8. WEATHER CONDITIONS AND PLANT ACTIVITIES DURING SAMPLING AT DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA[a]

Time	Temperature (°F)	Barometric pressure (mm Hg)	Wind Speed	Wind Direction	Plant activities
August 7					
1700	97	760	8	West	Normal
1800	96		12	West	Normal
1900	92		10	West	Normal
2000	87		10	West	Normal
2100	84	760	8	West	Normal
2200	82		8	West	Normal
2300	79		6	West	Normal
2400	80		2	West	Normal
August 8					
0100	74	760	0	-	Normal
0200	74		6	West	Normal
0300	74		8	West	Normal
0400	73		6	West	Normal
0500	71	760	4	West	Normal
0600	69		0	-	Normal
0700	67		0	-	Normal
0800	73		0	-	Normal
0900	83	760	0	-	Normal
1000	87		2	West	Incinerator feed rate reduced
1100	91		6	West	Normal
1200	96		2	Northwest	Normal
1300	98	760	4	Northwest	Normal
1400	101		4	West	Normal
1500	101		10	West	Possible event
1600	103		12	West	Normal
1700	102	759	12	West	Normal
1800	99		13	West	Normal

---

[a] No precipitation during sampling period.

E. I. du PONT de NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

PRESAMPLING SITE SURVEY

The presampling site survey at du Pont's Corpus Christi, Texas, plant was conducted on July 11, 1975. The following personnel were present:

Mr. Charles Evans	Plant Manager, du Pont
Mr. Dave Brooks	Assistant Plant Manager, du Pont
Mr. Phil Kuykendall	Midwest Research Institute

This du Pont plant is located on Corpus Christi Bay approximately 3 miles northeast of downtown Corpus Christi. The immediate area surrounding the plant contains several industries, but the small town of Ingleside is about 1 mile east of the plant. Wind direction in the summer is generally from the south, south southeast, and south southwest.

Carbon tetrachloride is produced by chlorination of methane or ethylene at elevated temperatures. All by-products are continuously recycled to insure total chlorination. Chlorine is obtained from an outside source. Solid wastes from the process are minimal and are not frequently removed from the reaction vessel.

Solid wastes are generally drummed and shipped to an outside firm for disposal although some wastes are dumped into du Pont's two landfills. One landfill is dedicated to the disposal of a mixture of cement, lime, and catalyst from Freon production. The other landfill normally receives such wastes as contaminated containers, spills, or "heavy ends" waste from the carbon tetrachloride production.

Wastewater is handled separately as process waste and storm runoff. Process wastes are channeled via open concrete ditches, to an equalization pond where the residence time is 3 to 6 days. Underground pipes discharge the wastewater into Lacita Channel at a flow rate of approximately 2,000 gal/min.

Storm runoff is discharged, via open dirt ditches, directly into Lacita Channel

FIELD SAMPLING

Field sampling at the du Pont plant was conducted on August 3, 1975. Air, soil, water, and sediment samples were collected. Detailed descriptions of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Due to equipment failure,only five air sampling stations were operated. Two were upwind and three were downwind. All samplers were operated for 24 hr utilizing one filter and two Tenax tubes in tandem. Sampling locations are shown in Figure A-6. Exact distance of each station to the production area was determined and reported along with other sampling data in Table A-9.

Soil Sampling

Soil sampling was conducted in three general areas:

S-1	Upwind (at Stations 1 and 2 on southern boundary)
S-2	Downwind (at Stations 3, 4, and 5 on northern boundary)
S-3	Landfill area (20 ft from edge of miscellaneous landfill)

All soil samples were composites.

Water Sampling

Water sampling was conducted at seven locations:

W-1	Raw plant water before use
W-2	Settling pond inlet (amberlite)
W-3	Settling pond inlet (grab)
W-4	Settling pond outlet (amberlite)
W-5	Settling pond outlet (grab)
W-6	Storm runoff outfall (grab)
W-7	Water standing in landfill

Sediment Sampling

Sediment sampling was conducted at four locations:

R-1	Settling pond inlet
R-2	Settling pond outlet
R-3	Storm runoff outfall

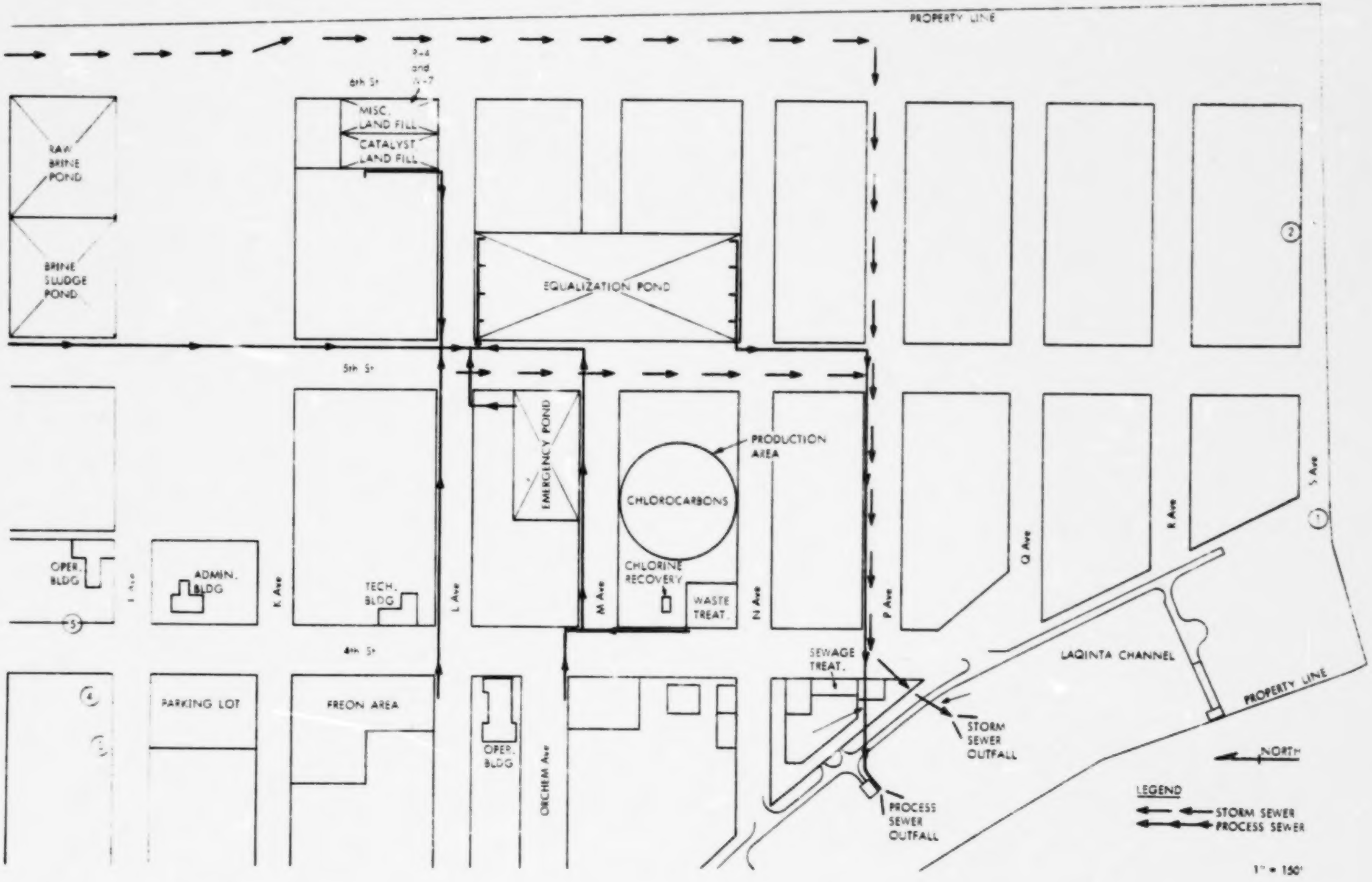

PROPERTY LINE
6th St
P-4
and
N-7
MISC.
LAND FILL
CATALYST
LAND FILL
RAW
BRINE
POND
BRINE
SLUDGE
POND
EQUALIZATION POND
2
5th St
EMERGENCY POND
PRODUCTION
AREA
CHLOROCARBONS
CHLORINE
RECOVERY
WASTE
TREAT.
S Ave
R Ave
Q Ave
P Ave
N Ave
M Ave
L Ave
K Ave
J Ave
1
OPER.
BLDG
ADMIN.
BLDG
TECH.
BLDG
5
4th St
4
3
PARKING LOT
FREON AREA
OPER.
BLDG
ORCHEM Ave
SEWAGE
TREAT.
LAQINTA CHANNEL
PROPERTY LINE
STORM
SEWER
OUTFALL
PROCESS
SEWER
OUTFALL
NORTH
LEGEND
STORM SEWER
PROCESS SEWER
1" = 150'

Figure A-6. Sample locations at E. I. du Pont de Nemours and Company, Inc., Corpus Christi, Texas

Table A-9. AIR SAMPLING DATA AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Upwind	1	675 ft south of production area	24 hr continuous	22.8	3.5	4,371	4
Upwind	2	700 ft south southeast of production area	24 hr continuous	19.5	3.5	3,621	4
Downwind	3	640 ft north northwest of production area	24 hr continuous	21.2	3.5	4,070	4
Downwind	4	64- ft north northwest of production area	24 hr continuous	21.2	3.5	4,007	4
Downwind	5	640 ft north northwest of production area	24 hr continuous	21.3	3.5	3,965	4

Plant Activities

Plant activities during sampling are shown in Tables A-10 and A-11, respectively.

Table A-10. PLANT ACTIVITIES DURING SAMPLING AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Time	Chlorocarbon unit	Landfill	Settling pond flow (gal/min)
August 3			
0700	Normal production	No activity	2,300
0800	Normal production	No activity	2,300
0900	Normal production	No activity	2,300
1000	Down	No activity	2,300
1100	Down	No activity	2,300
1200	Normal production	No activity	2,300
1300	Normal production	No activity	2,300
1400	Normal production	No activity	2,300
1500	Normal production	No activity	2,300
1600	Normal production	No activity	2,300
1700	Normal production	No activity	2,300
1800	Normal production	No activity	2,300
1900	Normal production	No activity	2,300
2000	Normal production	No activity	2,300
2100	Normal production	No activity	2,300
2200	Normal production	No activity	2,300
2300	Normal production	No activity	2,300
2400	Normal production	No activity	2,300
0100	Normal production	No activity	2,300
0200	Normal production	No activity	2,300
0300	Normal production	No activity	2,300
0400	Normal production	No activity	2,300
0500	Normal production	No activity	2,300
0600	Normal production	No activity	2,300
0700	Normal production	No activity	2,300
0800	Normal production	No activity	2,300
0900	Normal production	No activity	2,300
1000	Normal production	No activity	2,300

Table A-11. WEATHER CONDITIONS DURING SAMPLING AT E. I. DU PONT DE NEMOURS AND COMPANY, INC., CORPUS CHRISTI, TEXAS

Time	Temperature (°C)	Barometric pressure (mm Hg)	Precipitation	Wind Speed	Wind Direction
August 3					
0700	28	762	none	19	South
0800	28	762	none	17	South
0900	28	762	none	15	South
1000	29	762	none	11	South
1100	29	761	none	11	South southeast
1200	29	761	none	11	South southeast
1300	29	761	none	10	South southeast
1400	30	761	none	10	South southeast
1500	29	761	none	12	South southeast
1600	29	760	rain	22	North northwest
1700	27	760	rain	9	East
1800	28	760	none	10	East southeast
1900	28	760	none	10	Southeast
2000	28	760	none	9	Southeast
2100	28	760	none	9	Southeast
2200	27	762	none	7	Southeast
2300	27	762	none	10	Southeast
2400	27	762	none	10	South southeast
August 4					
0100	27	762	none	10	South southwest
0200	26	762	none	18	South
0300	28	761	none	16	South
0400	27	761	none	17	South
0500	28	761	none	17	South
0600	29	761	none	15	South
0700	29	761	none	15	South southwest
0800	29	760	none	18	South southwest
0900	30	760	none	15	South southwest
1000	30	760	none	13	South southwest
1100	30	760	rain	12	Southwest

DIAMOND SHAMROCK CORPORATION, DEERK PARK, TEXAS

PRESAMPLING SITE SURVEY

The presampling site survey at Diamond Shamrock's, Deer Park, Texas, plant was conducted on July 10, 1975. The following personnel were present:

Mr. Lavern R. Heble	Environment Control Manager, Gulf Coast Area, Diamond Shamrock Corporation
Mr. William C. Hutton	Senior Environmental Control Engineer, Diamond Shamrock Corporation
Mr. Bob Baxter	Perchloroethylene Unit Manager, Diamond Shamrock Corporation
Ms. Sandra Quinlivan	TRW, Rodondo Beach, California
Mr. Phil Kuykendall	Midwest Research Institute

This Diamond Shamrock plant is located in the heart of a huge industrial area along the Honston ship channel. The nearest residential area is Deer Park, located approximately 5 miles south of the plant. Wind direction in the summer months is mostly from the south.

Perchloroethylene and trichloroethylene are produced in this plant by the reaction of chlorine and hydrocarbons. The chlorine used is produced at a nearby Diamond Shamrock plant and is piped to the production area. "Hex" solids resulting from the process are stored in large tanks awaiting disposal.

The major possible sources of HCB and HCBD are (a) the production area and (b) the "Hex" solids storage area.

Diamond Shamrock uses two types of waste disposal; contracted solid waste disposal and channeling of wastewater into Patrick Bayou. The solid wastes are transferred from the holding tank to a tank truck which transports the waste to an outside firm for incineration or landfill disposal. Solid waste removal does not occur daily. A solvent flush of the lines into the tank truck follows each dumping of "Hex" solids. Wastewater is steam-stripped in the production area then piped to a waste treatment plant prior to discharge into Patrick Bayou.

FIELD SAMPLING

Field sampling at the Diamond Shamrock plant was conducted on August 20, 1975 after several postponements due to plant down time. Air, soil, and water samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight air sampling stations were used--three upwind and five downwind. To avoid possible breakthrough of the Tenax®-GC, smaller critical orifices were used. All samples were operated for three 8-hr periods, resulting in a 24-hr sampling time. Sampling locations are shown in Figure A-7. The exact distance of each station to the production area was determined and reported along with other sampling data in Table A-12.

Soil Sampling

Soil sampling was conducted at three areas:

S-1	Upwind (along southern boundary)
S-2	Downwind (along northern boundary)
S-3	Production area

All soil samples were composites.

Water Sampling

Water sampling was conducted at two points:

W-1	Incoming channel water
W-2	Process water outfall

All samples were grab samples.

No sediment sampling was conducted because of no existing sampling site.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-13. Plant activities were normal during the entire sampling period.

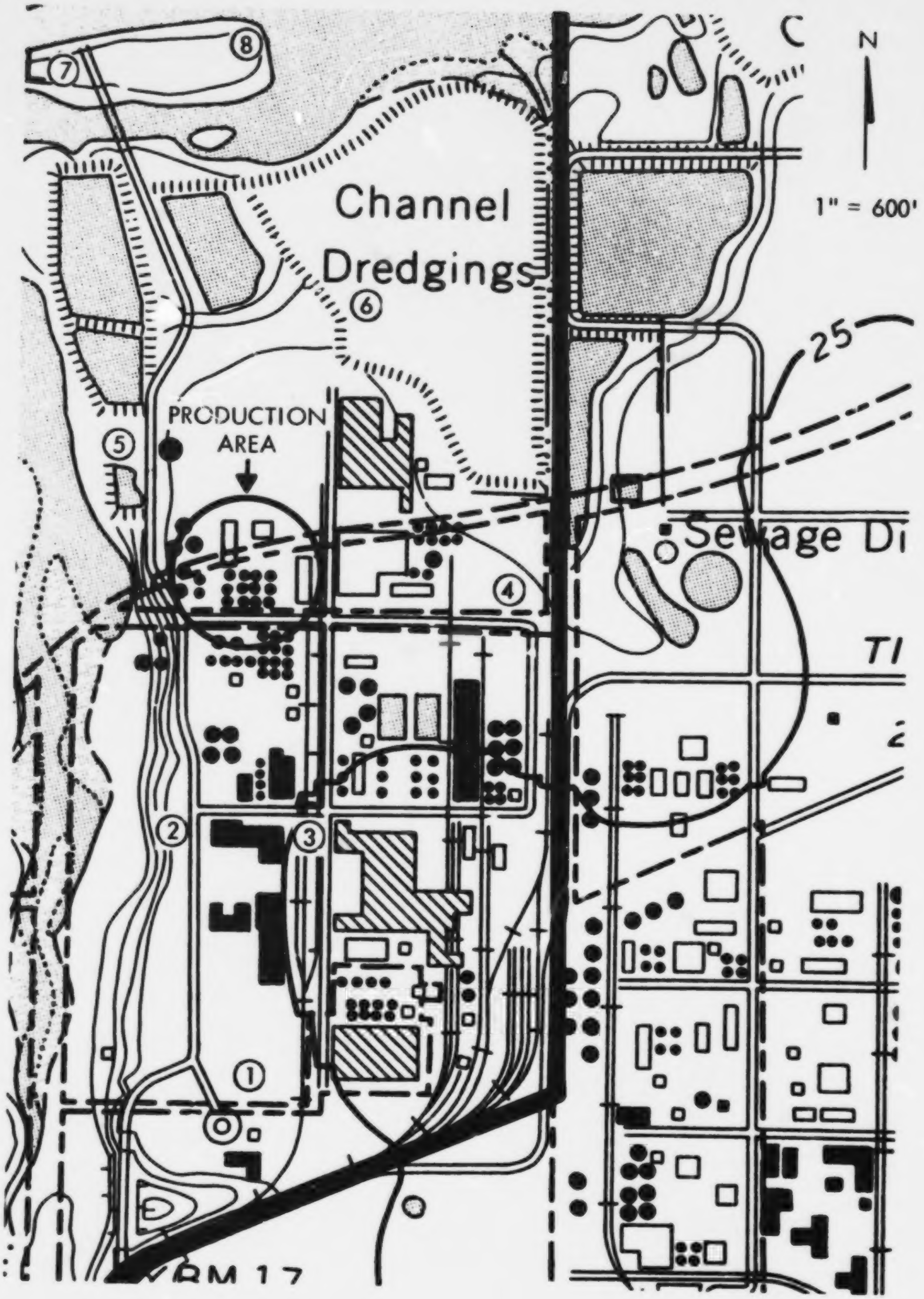

N
1" = 600'
Channel
Dredgings
PRODUCTION
AREA
Sewage Di
25
TI
FM 17
①
②
③
④
⑤
⑥
⑦
⑧

Figure A-7. Sampling locations at Diamond Shamrock Corporation, Deer Park, Texas

Table A-12. AIR SAMPLING DATA AT DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Far upwind	1	1,300 ft south of "Perc Plant" area	24 hr continuous	24.2	0.4	580	4
Near upwind	2	510 ft southwest of "Perc Plant" area	24 hr continuous	24.9	0.4	598	4
Near upwind	3	420 ft southeast of "Perc Plant" area	24 hr continuous	20.2	0.4	485	4
Near downwind	4	1,200 ft northeast of "Perc Plant" area	24 hr continuous	22.5	0.4	540	4
Near downwind	5	1,300 ft northwest of "Perc Plant" area	24 hr continuous	22.6	0.4	542	4
Near downwind	6	1,860 ft north northeast of "Perc Plant" area	24 hr continuous	25.3	0.4	608	4
Far downwind	7	2,850 ft north northwest of "Perc Plant" area	24 hr continuous	23.3	0.4	559	4
Far downwind	8	2,900 ft north of "Perc Plant" area	24 hr continuous	23.1	0.4	555	4

Table A-13. WEATHER CONDITIONS DURING SAMPLING AT DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Time	Temperature (°C)	Barometric pressure	Precipitation	Wind Speed	Wind Direction
August 20					
1600	34	766	None	4	Southeast
1700	34	766		5	South southeast
1800	33	766		4	South
1900	33	766		3	South southeast
2000	31	767		2	South southeast
2100	31	767		1	South
2200	29	767		1	South
2300	29	767		1	South
2400	27	767		1	South
August 21					
0100	26	766		1	South
0200	26	766		1	South
0300	28	766		1	North northwest
0400	28	766		1	North
0500	30	765		2	North northwest
0600	30	765		6	Southeast
0700	30	765		5	South
0800	30	765	Rain	3	North northwest
2100	28	765	Rain	1	East northeast
2200	28	765	None	1	East
2300	27	765		1	North northeast
2400	25	765		1	North
August 22					
0100	25	765		1	North northeast
0200	28	765		1	North
0300	29	766		1	West northwest
0400	29	766		1	East
0500	31	766		1	East
0600	31	766		1	East
0700	31	766		1	North northeast
0800	30	766		1	East northeast
0900	31	766		2	North northeast

CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

PRESAMPLING SITE SURVEY

The presampling site survey at Ciba-Geigy Corporation, St. Gabriel, Louisiana, plant was conducted on July 15, 1975. The following personnel were present:

Mr. William F. Snyder	Staff Engineer, Environmental Engineering, Ciba-Geigy
Mr. Phil Kuykendall	Midwest Research Institute

Ciba-Geigy Corporation is located in a large industrial area which is approximately 20 miles south of Baton Rouge. The plant is on the east bank of the Mississippi River in a predominantly swampy area with minimal residences. Wind direction during the summer months are generally from the south and west.

Triazine herbicides are produced by the amination of cyanuric chloride. The production processes result in the accumulation of "still bottoms" with an approximate concentration of 2,000 μg/g.

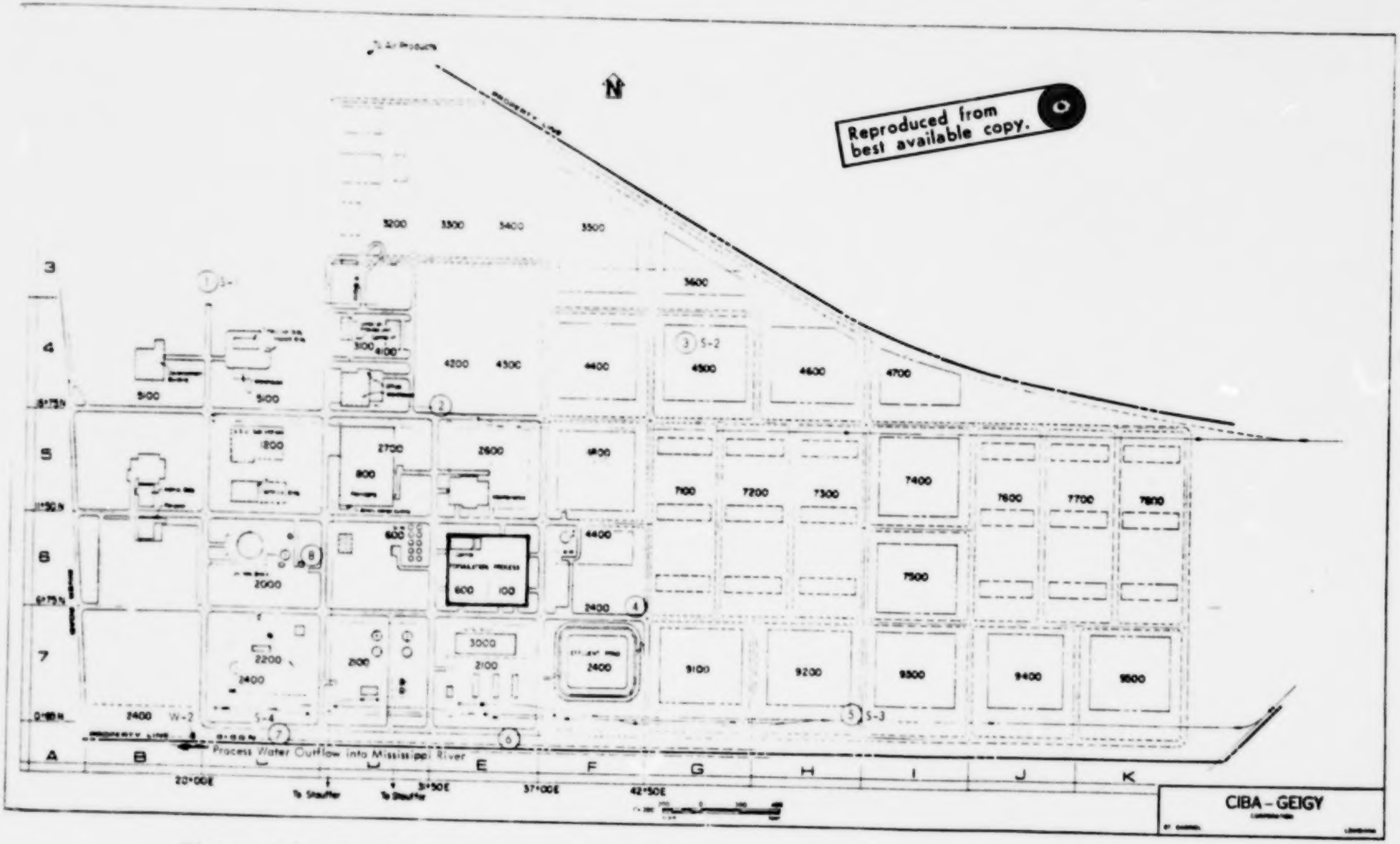
Solid wastes are drummed and shipped to an outside firm for incineration. A vent scrubber is used for vapor emissions. Wastewater is discharged via open ditches to a holding pond, then discharged into the Mississippi River at an average flow rate of 2,000 gal/min. An emergency wastewater outfall into Bayou Braud is occasionally used.

FIELD SAMPLING

Field sampling at the Ciba-Geigy plant was conducted on August 13, 1975. Air, soil, and water samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight air sampling stations were positioned around the plant because of the erratic wind direction. Only one Tenax®-GC tube was used. To avoid possible breakthrough of the Tenax®-GC, sampling was operated 2 hr out of every 8-hr period. The sampling locations are shown in Figure A-8. Exact distance of each station to the production areas was determined and reported along with other sampling data in Table A-14.


To Air Products
PROPERTY LINE
N
3200
3300
3400
3500
3600
3
4
5
6
7
① S-1
②
③ S-2
④
⑤ S-3
⑥
⑦
⑧
5100
4100
4200
4300
4400
4500
4600
4700
1200
2700
800
2600
6100
7100
7200
7300
7400
7600
7700
7800
600
100
7500
2000
2400
2200
2100
3000
9100
9200
9300
9400
9500
W-2
S-4
PROPERTY LINE
Process Water Outflow into Mississippi River
A
B
C
D
E
F
G
H
I
J
K
20+00E
To Stauffer
To Stauffer
31+50E
37+00E
42+50E
CIBA-GEIGY

Reproduced from best available copy.

Figure A-8. Sample locations at Ciba-Geigy Corporation, St. Gabriel, Louisiana

Table A-14. AIR SAMPLING AT CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
1	2,200 ft northwest of production area	1st 2 hr of 8 hr	7.6	3.5	1,772	4
2	920 ft north of production area	1st 2 hr of 8 hr	8.8	3.5	2,164	4
3	1,600 ft northeast of production area	1st 2 hr of 8 hr	6.8	3.5	1,630	4
4	800 ft east southeast of production area	1st 2 hr of 8 hr	6.3	3.5	1,442	4
5	2,200 ft southeast of production area	1st 2 hr of 8 hr	6.4	3.5	1,561	4
6	950 ft south of production area	1st 2 hr of 8 hr	Lost	3.5	Lost	4
7	1,600 ft southwest of production area	1st 2 hr of 8 hr	6.1	3.5	1,277	4
8	1,000 ft west of production area	1st 2 hr of 8 hr	5.6	3.5	1,298	4

Soil Sampling

Soil sampling was conducted at four locations:

S-1	Northwest of plant at Air Sampling Site No. 1
S-2	Northeast of plant at Air Sampling Site No. 3
S-3	Southeast of plant at Air Sampling Site No. 5
S-4	Southwest of plant at Air Sampling Site No. 7

All soil samples were composites.

Water Sampling

Water sampling was conducted at two locations:

W-1	Raw plant water before use
W-2	Process outflow

All water samples were grab samples.

Sediment Sampling

No sediment samples were taken. The process outflow ditch has a gravel bottom. The effluent pond was not available for sampling, per Ciba-Geigy's request.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-15. Plant activities were normal.

Table A-15. WEATHER CONDITIONS DURING SAMPLING AT CIBA-GEIGY, ST. GABRIEL, LOUISIANA

			Wind	
Time	Temperature (°C)	Precipitation	Speed	Direction
August 13				
1500	39	none	3	North northwest
1600	37	↓	7	Northwest
1700	37		5	Northwest
1800	36		5	North northwest
1900	36		4	Southwest
2000	36		7	South
2100	34		5	South
2200	32		5	South
2300	32		3	Southwest
2400	29		5	Southwest
August 14				
0100	27		5	West southwest
0200	27		6	Southwest
0300	27		8	Souwthwest
0400	29		7	West
0500	29		7	West
0600	30		5	West
0700	32		4	West northwest
0800	32		6	Northwest
0900	33		5	Northwest
1000	35		6	North northwest
1100	35		4	North northwest
1200	36		4	North northwest
1300	36		2	North northwest

OLIN CORPORATION, MCINTOSH, ALABAMA

PRESAMPLING SITE SURVEY

The presampling site survey at Olin Corporation's McIntosh, Alabama, plant was conducted on July 23, 1975. The following personnel were present:

Mr. J. Oertling	Works Manager, Olin
Mr. F. Champion	Production Manager, Organic Section, Olin
Mr. C. Hovater	Q. C. Manager, Olin
Mr. R. Reams	Technical Manager, Olin
Mr. N. Barone	Specialist - Environmental Affairs, Olin
Mr. D. Sauter	Midwest Research Institute

Olin Corporation is located approximately 30 miles north of Mobile, Alabama. The area in the immediate vicinity of the plant is flat and marshy. Residential areas around the plant are minimal. Winds are generally from the south during the summer.

The production of pentachloronitrobenzene is by chlorination and nitration of isomeric chlorobenzenes. The production process results in the accumulation of "still bottoms" which are cast into 27-ft^3 blocks containing 80 to 90% HCB. The rated capacity of the plant for PCNB is approximately 7 million pounds per year. Approximately 2.8 to 3.0 million pounds per year of HCB is generated in this process.

Chlorine is also produced by mercury cells using carbon electrodes at a rate of 130,000 tons/year.

Solid wastes (HCB blocks) are stored in an open field in the southeast corner of the plant. The HCB block pile is covered with plastic. This pile represents HCB wastes from the last 2-1/2 years of PCNB production. Ultimately, Olin hopes to reclaim the HCB.

Wastewater is discharged into two open ditches with an average combined flow of 5 million gallons per day. The south ditch, which is adjacent to the PCNB plant and receives runoff from the waste disposal settling pond, contributes mostly to this flow. Both ditches combine outside the plant boundary, and flow into a basin, and ultimately into the Tombigbee River.

At least two landfills were observed. The first landfill is located directly outside the northeast corner of the plant boundary. This landfill was used for HCB disposal before 1971. The second landfill is the southeastern section of the plant is primarily a garbage dump. No landfills are currently in use for chemical disposal.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of August 18, 1975.

FIELD SAMPLING

Field sampling at the Olin plant was conducted on August 18, 1975. Air, soil, water, and sediment samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Eight sampling stations were positioned to give two upwind, three midplant, and three downwind sites. Two Tenax®-GC sampling tubes were operated in tandem at each site for three, 3-hr periods. Sampling locations are shown in Figure A-9. The exact distance of each station to the production area was determined and reported along with other sampling data in Table A-16.

Soil Sampling

Composite grab samples were taken outside and inside the plant boundary:

S-1	Downwind (north boundary)
S-2	Old landfill (northeast boundary)
S-3	Brine pond area
S-4	Center road (running north/south)
S-5	High lift route (organic plant to storage area)
S-6	Southeast landfill
S-7	"Hex" storage area
S-8	Old "Hex" dump area
S-9	East road

Reproduced from best available copy.

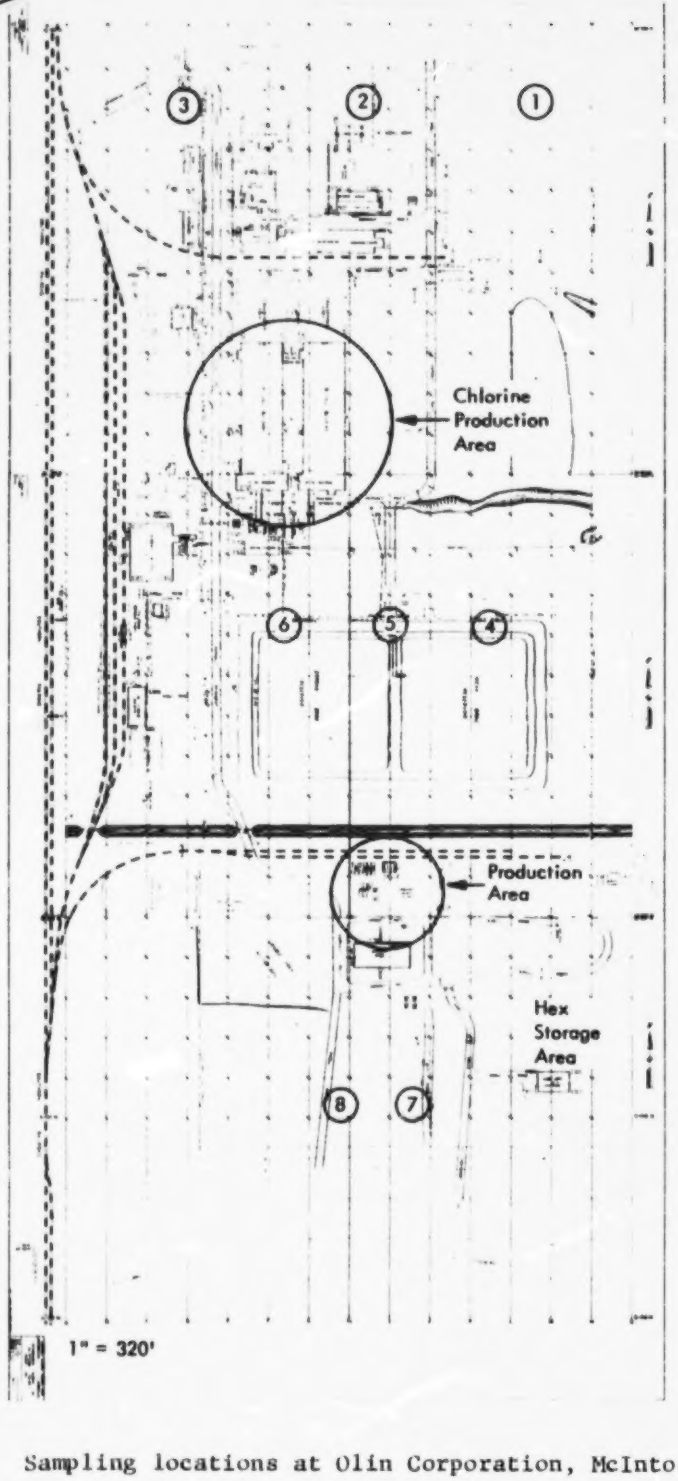

③
②
①
Chlorine Production Area
⑥
⑤
④
Production Area
Hex Storage Area
⑧
⑦
1" = 320'

Figure A-9. Sampling locations at Olin Corporation, McIntosh, Alabama

Table A-16. AIR SAMPLING DATA AT OLIN CORPORATION, MCINTOSH, ALABAMA

General area	Sample No.	Exact location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
Far downwind	1	2,100 ft north northeast of production area	1st 3 hr of 12 hr	10.0	3.5	2,103	4
Far downwind	2	2,100 ft north of production area	1st 3 hr of 12 hr	5.2	3.5	1,100	4
Far downwind	3	2,200 ft north northwest of production area	1st 3 hr of 12 hr	5.7	3.5	1,204	4
Near downwind	4	720 ft north northeast of production area	1st 3 hr of 12 hr	6.9	3.5	1,445	20[a]
Near downwind	5	700 ft north of production area	1st 3 hr of 12 hr	7.0	3.5	1,473	20[a]
Near downwind	6	800 ft north northwest of production area	1st 3 hr of 12 hr	7.0	3.5	1,472	20[a]
Upwind	7	640 ft south of production area	1st 3 hr of 12 hr	7.8	3.5	1,630	4
Upwind	8	640 ft south of production area	1st 3 hr of 12 hr	7.3	3.5	1,525	4

---

[a] Samplers were positioned on Brine Pond dike.

S-10	West road
S-11	Upwind south road

Water Sampling

The following grab samples were obtained:

W-1	Weak brine pond
W-2	Strong brine pond
W-3	Settling pond
W-4	North/south running ditch
W-5	Southern ditch area (upper drop)
W-6	Combined creek (200 yard before basin)
W-7	Basin (at mouth of creek)
W-8	24-Hr composite of plant effluent (combined creek)
W-9	Solar pond, west
W-10	Solar pond, east

Sediment Sampling

One sediment sample was collected at the strong brine pond.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-17. Plant activities were normal.

Table A-17. WEATHER CONDITIONS DURING SAMPLING AT OLIN CORPORATION, MCINTOSH, ALABAMA

Time	Temperature (°C)	Barometric pressure (mm Hg)	Wind Speed	Wind Direction	Precipitation
August 18					
1500[a]	37	738	2-4	South	None
1600[a]	37	738	2-4	Southeast	None
1700[a]	36	738	4-10	North northeast	None
1800			6-13	North	Rain
1900			4-6	Southeast	None
2000			2-6	East	None
2100			2-4	Northwest	None
2200			2-4	West	None
2300			-	No wind	None
2400[a]	26	756	2-4	South	None
August 19					
0100[a]	23	741	2-4	Southwest	None
0200[a]	23	740	-	No wind	None
0300[a]	23	740	-	No wind	None
0400			-	No wind	None
0500			-	No wind	None
0600			-	No wind	None
0700			2-4	North northeast	None
0800[a]	25	740	-	No wind	None
0900[a]	27	740	-	No wind	None
1000[a]	29	739	-	No wind	None
1100[a]	32	739	-	No wind	None
1200[a]	35	738	2-6	North	None

---

[a] Indicates air sampling.

KAISER ALUMINUM AND CHEMICAL CORPORATION, GRAMERCY, LOUISIANA

The presampling site survey at Kaiser Aluminum was conducted on August 14, 1975. The following personnel were present:

Dr. Robert M. Hansen	Research and Development, Kaiser
Mr. Phil Fourmet	Environmental Manager, Kaiser
Mr. Bob Curtis	Environmental Control Specialist, Kaiser
Mr. Phil Kuykendall	Midwest Research Institute

Upon discussion with the plant officials, it was learned that chlorine production utilizing graphite anodes had been terminated in 1973, and was replaced by dimensionally stabilized anodes. For waste disposal, prior to 1973, solid wastes were dispoded by landfill. These residues have since been covered by aluminum production wastes. Water effluent is channeled into the Mississippi river following on-line pH adjustment.

As a result of the change in production technology and the solid wastes disposed since 1973, it was decided that sampling at this plant would yield no usable data. Furthermore, samples collected from the Linden Chlorine Company, at Linden, New Jersey, and the other chlorohydrocarbon plants, which also produce chlorine, would serve the purpose of monitoring the chlorine production plant for the HCB and HCBD emission.

PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

PRESAMPLING SITE SURVEY

The presampling site survey at PPG Industries' Lake Charles, Louisiana, plant was conducted on August 22, 1975. The following personnel were present:

Mr. T. G. Taylor	Technical Plant Manager, PPG
Mr. Thomas C. Jeffery	Chief Process Engineer, PPG
Dr. Earl Gorton	Senior Research Supervisor, Organics, PPG
Mr. C. A. Burns	Environmental Control Specialist, PPG
Mr. Mark Wood	Environmental Analysis Coordinator, PPG
Dr. Raymond Li	Midwest Research Institute

PPG Industries is located more than 2 miles west of downtown Lake Charles, Louisiana. The surrounding terrain is flat and marshy. The PPG canal runs through the plant and into the Calcasieu River which flows to Lake Charles. The closest residential area is about 1 mile northwest of the plant. The wind, in the summer months, is very variable but most likely from south southeast and least likely from the west.

Chemicals produced in this plant include trichloroethylene, perchloroethylene, ethylene dichloride, ethyl chloride, vinylidene chloride, methyl chloroform, vinyl chloride, chlorine, hydrochloric acid, caustic soda, and silica pigments. The current production capacities of trichloro- and perchloroethylene are 350 tons/day. However, the production of 725 tons/day could be achieved and was produced in the past. The plant operates 24 hr a day, 7 days a week.

Trichloro- and perchloroethylene are produced by a catalytic oxychlorination process rather than the thermal process of chlorine and hydrocarbons, thus resulting at a lower reaction temperature. The chlorine is produced in the plant (DSA has been used since 1969). The production wastes are piped into the incinerator and burnt at a residence time from 1/4 to 1/3 sec at 2500°F. The water effluent is channeled into the PPG canal which flows into the Calcasieu River. The PPG canal also received runoffs from the organochlorine production as well as effluents from the power plant.

Prior to the operation of the incinerator, landfill was used for waste disposal. The old landfill site was covered with water. It is still being used for wastes than cannot be burnt in the incinerator or when incinerator breakdown occurs.

At the conclusion of the presampling survey, it was agreed upon that field sampling would be tentatively scheduled in the week of September 2, 1975.

FIELD SAMPLING

Field sampling at the PPG plant was conducted on September 4, 1975. Air, soil, water, and sediment samples were collected. Detailed description of the sampling, plant activities, and weather conditions, are discussed below.

Air Sampling

Ten sampling stations were positioned to encircle the plant's incinerator and organochlorine production area. Two Tenax-GC® sampling tubes were operated in tandem with 24-hr continuous sampling time. Sampling locations are shown in Figure A-10. The exact distance of each station to the incinerator/production area is listed along with other sampling data in Table A-18.

Soil Sampling

Composite grab samples were taken outside and inside the plant area.

S-1	Landfill composite
S-2	Composite at Air Stations 7, 5, and 4
S-3	Composite at Air Stations 8, 9, and 10
S-4	Composite near Air Station 1 on Columbia Southern Road

Water Sampling

Grab samples were obtained at the following locations:

W-1	Incinerator feed water, lake water
W-2	Scrubber water
W-3	Inlet treatment canal organic effluent before scimmer
W-4	Outlet treatment canal organic effluent after scimmer.
W-5	Surface water, landfill
W-6	Downstream PPG canal, at Mobile Bridge No. 1, 1 gal. taken
W-7	Ship channel, adjacent to Air Station No. 10

Sediment Sampling

Sediment samples were collected at three general areas.

R-1	Downstream PPG canal near Air Sampling Station No. 1
R-2	Main organic plant effluent, near Air Sampling Station No. 2
R-3	PPG ship channel, near Air Sampling Station No. 10

Reproduced from best available copy.

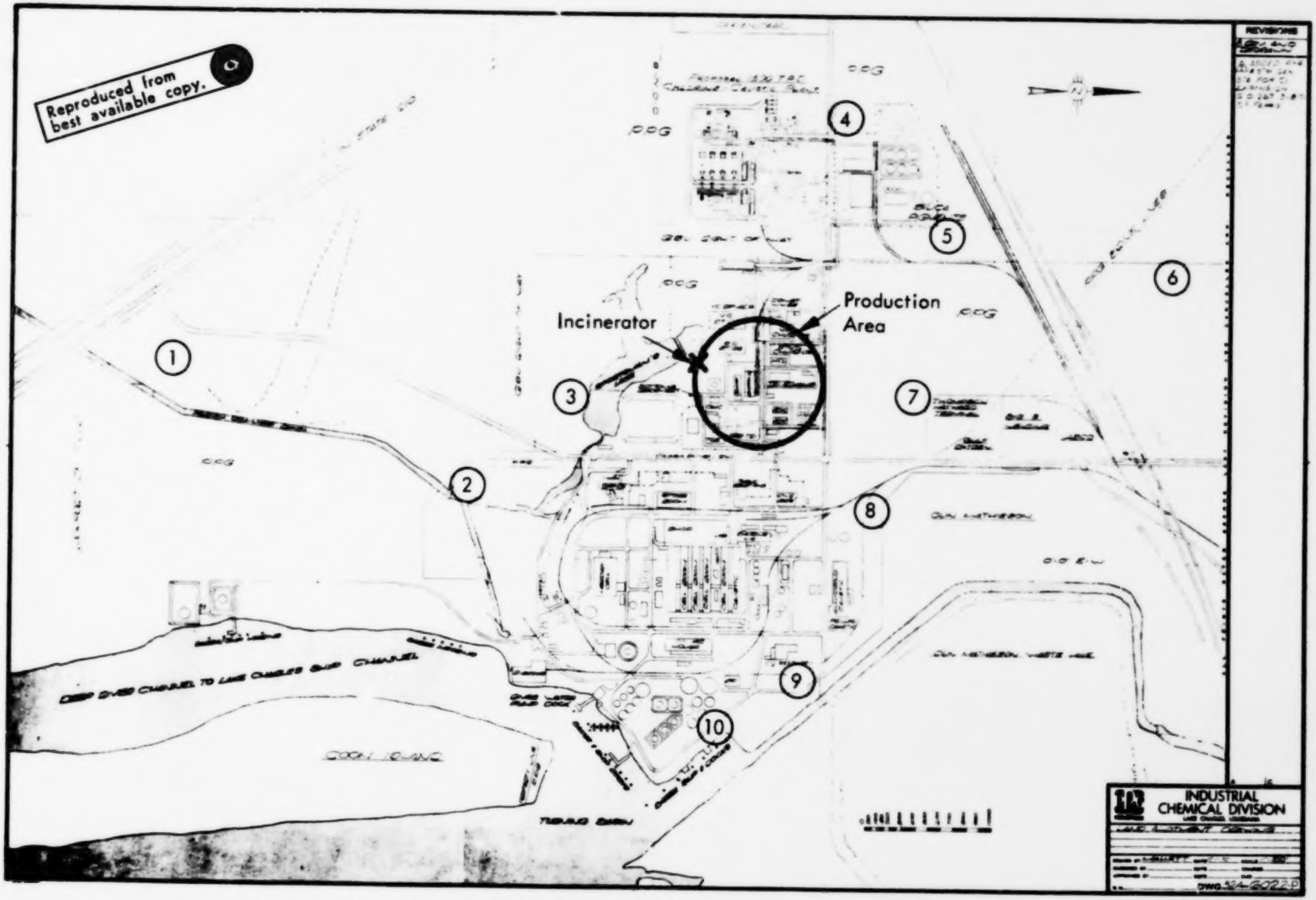

1
2
3
4
5
6
7
8
9
10
Incinerator
Production Area
PPG
DEEP RIVER CHANNEL TO LAKE CHARLES SHIP CHANNEL
COON ISLAND
TURNING BASIN
OLIN MATHIESON
OLIN MATHIESON WASTE LAKE
REVISIONS
INDUSTRIAL CHEMICAL DIVISION
DWG 324-2022-D

Figure A-10. Sampling locations at PPG Industries, Lake Charles, Louisiana

Table A-18. AIR SAMPLING DATA AT PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sample No.[a]	Location	Sampling period	Total sampling time (hr)	Sampling rate (ℓ/min)	Sample vol. (ℓ)	Sampler height (ft)
1	4,700 ft south of production area	24 hr continuous	21.6	0.9	1,180	4
2	2,500 ft south southeast of production area	24 hr continuous	21.4	0.9	1,170	4
3	1,550 ft south of production area	24 hr continuous	21.3	0.9	1,210	4
4	2,300 ft west northwest of production area	24 hr continuous	21.0	0.9	1,170	4
5	2,000 ft northwest of production area	24 hr continuous	19.0[b]	0.9	950[b]	4
6	3,500 ft north northwest of production area	24 hr continuous	22.0	0.9	1,250	4
7	1,250 ft north of production area	24 hr continuous	20.7	0.9	1,180	4
8	1,250 ft northeast of production area	24 hr continuous	21.8	0.9	1,190	4
9	2,250 ft east of production area	24 hr continuous	21.9	0.9	1,250	4
10	2,700 ft east southeast of production area	24 hr continuous	21.9	0.9	1,130	4

---

[a] Stations were positioned surrounding the production area.

[b] Approximate value due to pump failure, indicates minimum volume.

Plant Activities and Weather Conditions

The weather conditions during sampling are shown in Table A-19. Plant activities were normal.

Table A-19. WEATHER CONDITIONS DURING SAMPLING AT PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Time	Temperature (°F)	Barometeric pressure (mm Hg)	Wind Speed (mph)	Wind Direction
Spetember 4				
1100	85	763	7	East
1200	86	762	7	East
1300	88	762	7	East
1400	85	762	7	East
1500	85	762	7	East
1600	85	762	7	East southeast
1700	82	762	8	East southeast
1800	80	762	7	East
1900	78	763	5	East
2000	77	763	4	East
2100	76	763	3	East
2200	75	763	2	West
2300	76	762	-	-
2400	75	762	-	-
September 5				
0100	75	762	3	East
0200	75	762	6	East
0300	75	762	5	East
0400	75	762	3	East
0500	75	762	2	East
0600	75	763	3	East
0700	74	763	2	East
0800	73	763	3	East
0900	72	763	3	East[a]

---

[a] Rain.

APPENDIX B

ANALYTICAL DATA

Table B-1. HCBD CONCENTRATIONS IN AIR SAMPLES FROM VULCAN MATERIALS COMPANY, WICHITA KANSAS

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCBD Total ng	HCBD µg/m³ a/
1	1935-2035	26	Filter	< 1	16.4
			Tenax	425	
	0120-0227	42	Filter	< 1	13.3
			Tenax	560	
	0430-0523	33	Filter	< 1	6.4
			Tenax	210	
	0841-0941	37	Filter	< 1	5.0
			Tenax	185	
	1320-1424	40	Filter	< 1	3.4
			Tenax	135	
2	1935-2035	17	Filter	< 1	11.9
			Tenax	200	
	0120-0227	36	Filter	< 1	12.6
			Tenax	455	
	0430-0523	29	Filter	< 1	28.4
			Tenax	825	
	0841-0941	32	Filter	< 1	6.4
			Tenax	205	
	1320-1424	35	Filter	< 1	10.6
			Tenax	370	
3	1935-	Lost	Filter		
			Tenax		
	0120-0227	57	Filter	< 1	5.4
			Tenax	310	
	0430-0523	45	Filter	< 1	4.1
			Tenax	185	
	0841-0941	51	Filter	< 1	3.0
			Tenax	150	
	1320-1424	54	Filter	< 1	10.1
			Tenax	545	
4	1945-2045	27	Filter	< 1	57
			Tenax	1,550	
	0110-0220	32	Filter	< 1	65
			Tenax	2,083	
	0450-0555	30	Filter	< 1	65
			Tenax	1,938	
	0902-1010	31	Filter	< 1	52
			Tenax	1,600	
	1307-1425	36	Filter	< 1	49
			Tenax	1,750	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCBD Total ng	 µg/m³
5	1945-2045	41	Filter	< 1	17
			Tenax	700	
	0110-0220	48	Filter	ND	18
			Tenax	850	
	0450-0555	44	Filter	< 1	51
			Tenax	2,250	
	0902-1010	46	Filter	< 1	36
			Tenax	1,650	
	1307-1425	53	Filter	< 1	< 0.02
			Tenax	< 1	
6	1945-2045	34	Filter	< 1	20
			Tenax	695	
	0110-0220	40	Filter	< 1	9
			Tenax	345	
	0450-0555	37	Filter	< 1	25
			Tenax	928	
	0902-1010	39	Filter	< 1	44
			Tenax	1,712	
	1307-1425	45	Filter	< 1	463
			Tenax	20,867	
7	1950-2050	148	Filter	< 1	3.2
			Tenax	475	
	0051-0158	166	Filter	< 1	3.6
			Tenax	590	
	0456-0555	146	Filter	< 1	2.8
			Tenax	417	
	0904-1010	163	Filter	< 1	11.4
			Tenax	1,862	
	1308-1425	190	Filter	< 1	17
			Tenax	3,250	
8	1950-2050	212	Filter	< 1	2.4
			Tenax	510	
	0103-0202	209	Filter	< 1	0.7
			Tenax	146	
	0458-0600	220	Filter	< 1	8.4
			Tenax	1,850	
	0908-1010	227	Filter	< 1	19.2
			Tenax	4,375	
	1308-1420	255	Filter	< 1	14.2
			Tenax	3,625	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCBD Total ng	HCBD $\mu g/m^3$
9	1950-2050	227	Filter	< 1	0.3
			Tenax	63	
	0103-0202	223	Filter	< 1	0.4
			Tenax	83	
	0458-0600	234	Filter	< 1	2.1
			Tenax	490	
	0908-1010	242	Filter	< 1	16
			Tenax	3,860	
	1308-1420	272	Filter	< 1	17
			Tenax	4,631	
10	1910-2310	809	Filter	< 1	12.2
			Tenax	9,850	
	0005-0330	691	Filter	< 1	6.5
			Tenax	4,500	
	0340-0728	768	Filter	< 1	2.6
			Tenax	2,000	
	0737-1117	741	Filter	< 1	1.6
			Tenax	1,200	
	1124-1433	637	Filter	< 1	0.7
			Tenax	438	
11	1910-2310	856	Filter	< 1	12.1
			Tenax	10,350	
	0005-0330	732	Filter	< 1	6.5
			Tenax	4,750	
	0340-0728	814	Filter	< 1	2.4
			Tenax	1,950	
	0737-1117	785	Filter	< 1	1.4
			Tenax	1,090	
	1124-1433	675	Filter	Sample lost	0.7
			Tenax	408	
12	1919-2315	863	Filter	< 1	20
			Tenax	17,500	
	2350-0315	738	Filter	< 1	22
			Tenax	16,333	
	0325-0712	817	Filter	< 1	6
			Tenax	4,500	
	0721-1101	792	Filter	< 1	5
			Tenax	4,000	
	1113-1433	720	Filter	< 1	3
			Tenax	1,833	

Table B-1. (continued)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCBD	
				Total ng	µg/m³
13	1915-2315	917	Filter	< 1	19
			Tenax	17,000	
	2350-0315	784	Filter	< 1	22
			Tenax	17,000	
	0325-0712	867	Filter	< 1	4
			Tenax	3,500	
	0721-1101	840	Filter	< 1	4
			Tenax	3,000	
	1113-1433	764	Filter	< 1	2
			Tenax	1,400	
14	1920-2259	806	Filter	< 1	0.004
			Tenax	3.5	
	2335-0258	816	Filter	< 1	0.2
			Tenax	150	
	0310-0640	844	Filter	< 1	0.03
			Tenax	25	
	0650-1046	949	Filter	< 1	0.1
			Tenax	130	
	1057-1435	876	Filter	< 1	4.2
			Tenax	3,642	
15	1920-2259	837	Filter	< 1	0.02
			Tenax	13.5	
	2335-0258	805	Filter	< 1	0.2
			Tenax	160	
	0310-0640	832	Filter	< 1	0.1
			Tenax	46	
	0650-1046	935	Filter	< 1	0.2
			Tenax	164	
	1057-1435	863	Filter	< 1	3.8
			Tenax	3,300	
16	2005-0010	862	Filter	< 1	0.2
			Tenax	133	
	0025-0345	702	Filter	< 1	0.04
			Tenax	30	
	0355-0744	804	Filter	< 1	0.1
			Tenax	48	
	0752-1154	849	Filter	ND	ND
			Tenax	ND	
	1200-1430	527	Filter	ND	0.1
			Tenax	29	

Table B-1. (concluded)

Sampling station	Sampling time	Volume sampled (liter)	Type of sample	HCBD	
				Total ng	µg/m³
17	2005-0010	730	Filter	ND	0.003
			Tenax	2	
	0025-0345	596	Filter	ND	0.008
			Tenax	5	
	0355-0744	682	Filter	ND	0.007
			Tenax	5	
	0752-1154	721	Filter	ND	0.01
			Tenax	9	
	1200-1430	447	Filter	ND	0.02
			Tenax	9	
18	2010-	548	Filter	< 1	0.2
			Tenax	130	
	0022-0355	711	Filter	< 1	0.1
			Tenax	54	
	0400-0755	785	Filter	< 1	0.1
			Tenax	61	
	0759-1121	858	Filter	< 1	0.1
			Tenax	123	
	1215-1430	451	Filter	< 1	0.1
			Tenax	39	
Tenax® GC blank					ND
Millipore filter blank					ND

---

a/ Concentration based on the sum of ng found on the filter and Tenax.
b/ ND - None detected.

Table B-2. HCBD CONCENTRATIONS IN AIR SAMPLES FROM STAUFFER CHEMICAL COMPANY, LOUISVILLE, KENTUCKY

Sampling station	Sampling time	Type of sample	Volume sampled (liter)	Concentration ($\mu g/m^3$) HCBD
1	1000-1400	Tenax®-GC	415	0.01
	1400-1800	Tenax®-GC	414	0.01
	1800-2200	Tenax®-GC	540	0.06
	2200-0200	Tenax®-GC	390	0.06
	0200-0600	Tenax®-GC	380	0.03
	0600-1000	Tenax®-GC	449	0.02
		Filters		ND
2	1000-1400	Tenax®-GC	455	0.04
	1400-1800	Tenax®-GC	408	0.06
	1800-2200	Tenax®-GC	514	0.02
	2200-0200	Tenax®-GC	463	0.03
	0200-0600	Tenax®-GC	336	0.01
	0600-1000	Tenax®-GC	450	0.01
		Filters		ND
3	1000-1400	Tenax®-GC	384	0.14
	1400-1800	Tenax®-GC	438	0.11
	1800-2200	Tenax®-GC	490	0.06
	2200-0200	Tenax®-GC	448	1.84
	0200-0600	Tenax®-GC	Lost	-
	0600-1000	Tenax®-GC	461	0.11
		Filters		ND
4	1000-1400	Tenax®-GC	432	6.42
	1400-1800	Tenax®-GC	456	5.13
	1800-2200	Tenax®-GC	454	5.75
	2200-0200	Tenax®-GC	519	9.32
	0200-0600	Tenax®-GC	437	5.57
	0600-1000	Tenax®-GC	470	1.24
		Filters		ND
5	1000-1400	Tenax®-GC	420	10.71
	1400-1800	Tenax®-GC	470	6.65
	1800-2200	Tenax®-GC	540	8.22
	2200-0200	Tenax®-GC	496	4.36
	0200-0600	Tenax®-GC	426	2.60
	0600-1000	Tenax®-GC	435	2.33
		Filters		ND

Table B-2. (concluded)

Sampling station	Sampling time	Type of sample	Volume sampled (liter)	Concentration ($\mu g/m^3$) HCBD
6	1000-1400	Tenax®-GC	408	5.82
	1400-1800	Tenax®-GC	455	1.83
	1800-2200	Tenax®-GC	464	10.56
	2200-0200	Tenax®-GC	442	3.92
	0200-0600	Tenax®-GC	425	2.65
	0600-1000	Tenax®-GC	468	1.09
		Filters		ND
7	1000-1400	Tenax®-GC	450	0.24
	1400-1800	Tenax®-GC	472	0.17
	1800-2200	Tenax®-GC	563	0.21
	2200-0200	Tenax®-GC	469	0.46
	0200-0600	Tenax^R-GC	426	0.46
	0600-1000	Tenax®-GC	470	1.27
		Filters		ND
8	1000-1400	Tenax®-GC	450	0.15
	1400-1800	Tenax®-GC	488	0.18
	1800-2200	Tenax®-GC	554	0.06
	2200-0200	Tenax®-GC	476	0.19
	0200-0600	Tenax®-GC	436	0.10
	0600-1000	Tenax®-GC	468	0.15
		Filters		ND
9	1000-1400	Tenax®-GC	455	0.04
	1400-1800	Tenax®-GC	492	0.02
	1800-2200	Tenax®-GC	562	0.02
	2200-0200	Tenax®-GC	483	0.14
	0200-0600	Tenax®-GC	466	0.05
	0600-1000	Tenax®-GC	468	0.11
		Filters		ND

Table B-3. HCBD CONCENTRATIONS IN AIR SAMPLES FROM DOW CHEMICAL COMPANY, PITTSBURG, CALIFORNIA

Sampling station	Type of sample	Volume sampled (liter)	Concentration (μg/m^3)
			HCBD
1	Tenax®GC (front)	4,336	< 0.01
	Tenax®GC (back)		< 0.01
	Filter		ND
2	Tenax®GC (front)	427	ND
	Tenax®GC (back)		ND
	Filter		ND
3	Tenax®GC (front)	4,166	0.1
	Tenax®GC (back)		0.1
	Filter		ND
4	Tenax®GC (front)	3,870	0.96
	Tenax®GC (back)		1.03
	Filter		ND
5	Tenax®GC (front)	3,713	0.4
	Tenax®GC (back)		0.3
	Filter		ND
6	Tenax®GC (front)	4,314	0.02
	Tenax®GC (back)		0.02
			ND
7	Tenax®GC (front)	962	0.3
	Tenax®GC (back)		ND
	Filter		ND
8	Tenax®GC (front)	3,963	0.03
	Tenax®GC (back)		0.03
	Filter		ND

Note: ND = none detected.

Table B-4. HCBD CONCENTRATIONS IN AIR SAMPLES FROM E. I. DU PONT DE NEMOURS AND COMPANY, CORPUS CHRISTI, TEXAS

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m^3) HCBD
1	Tenax®-GC, front	4,371	0.013
	Tenax®-GC, back	4,371	0.009
	Filter	4,371	ND
2	Tenax®-GC, front	3,621	0.002
	Tenax®-GC, back	3,621	0.001
	Filter	3,621	ND
3	Tenax®-GC, front	4,070	0.015
	Tenax®-GC, back	4,070	0.012
	Filter	4,070	ND
4	Tenax®-GC, front	4,007	0.017
	Tenax®-GC, back	4,007	0.012
	Filter	4,007	ND
5	Tenax®-GC, front	3,965	0.018
	Tenax®-GC, back	3,965	0.016
	Filter	3,965	ND

Table B-5. HCBD CONCENTRATIONS IN AIR SAMPLES FROM DIAMOND SHAMROCK CORPORATION, DEER PARK, TEXAS

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m^3) HCBD
1	Tenax®-GC, front	580	2.44
	Tenax®-GC, back	580	ND
	Filter	580	ND
2	Tenax®-GC, front	598	0.09
	Tenax®-GC, back	598	ND
	Filter	598	ND
3	Tenax®-GC, front	485	0.15
	Tenax®-GC, back	485	ND
	Filter	485	ND
4	Tenax®-GC, front	540	0.12
	Tenax®-GC, back	540	ND
	Filter	540	ND
5	Tenax®-GC, front	542	1.80
	Tenax®-GC, back	542	ND
	Filter	542	ND
6	Tenax®-GC, front	608	0.19
	Tenax®-GC, back	608	ND
	Filter	608	ND
7	Tenax®-GC, front	559	0.20
	Tenax®-GC, back	559	ND
	Filter	559	ND
8	Tenax®-GC, front	555	0.14
	Tenax®-GC, back	555	ND
	Filter	555	ND

Table B-6. HCBD CONCENTRATIONS IN AIR SAMPLES FROM CIBA-GEIGY CORPORATION, ST. GABRIEL, LOUISIANA

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration ($\mu g/m^3$) HCBD
1	Tenax®-GC	1,772	0.025
	Filter	1,772	ND
2	Tenax®-GC	2,164	0.028
	Filter	2,164	ND
3	Tenax®-GC	1,630	ND
	Filter	1,630	ND
4	Tenax®-GC	1,442	0.017
	Filter	1,442	ND
5	Tenax®-GC	1,561	0.019
	Filter	1,561	ND
6	Lost	Lost	Lost
7	Tenax®-GC	1,277	ND
	Filter	1,277	ND
8	Tenax®-GC	1,298	0.096
	Filter	1,298	ND

Table B-7. HCBD CONCENTRATIONS IN AIR SAMPLES FROM PPG INDUSTRIES, LAKE CHARLES, LOUISIANA

Sampling station	Type of sample	Volume sampled (ℓ)	Concentration (μg/m³) HCBD
1	Tenax®-GC, front	1,180	0.03
	Tenax®-GC, back	1,180	ND
	Filter	1,180	ND
2	Tenax®-GC, front	1,170	0.09
	Tenax®-GC, back	1,170	ND
	Filter	1,170	ND
3	Tenax®-GC, front	1,210	0.04
	Tenax®-GC, back	1,210	ND
	Filter	1,210	ND
4	Tenax®-GC, front	1,170	0.18
	Tenax®-GC, back	1,170	0.02
	Filter	1,170	ND
5	Tenax®-GC, front	950	ND
	Tenax®-GC, back	950	ND
	Filter	950	ND
6	Tenax®-GC, front	1,250	0.17
	Tenax®-GC, back	1,250	ND
	Filter	1,250	ND
7	Tenax®-GC, front	1,180	ND
	Tenax®-GC, back	1,180	ND
	Filter	1,180	ND
8	Tenax®-GC, front	1,190	ND
	Tenax®-GC, back	1,190	ND
	Filter	1,190	ND
9	Tenax®-GC, front	1,250	0.16
	Tenax®-GC, back	1,250	ND
	Filter	1,250	ND
10	Tenax®-GC, front	1,130	0.48
	Tenax®-GC, back	1,130	0.07
	Filter	1,130	ND

APPENDIX C

METHOD DEVELOPMENT FOR SAMPLING AND ANALYSIS

LITERATURE SEARCH

A search of the literature to 1967 revealed that there was no specific method for sampling HCBD in water. The method used most often for sampling pollutants in water was the "grab technique." Detailed procedures of the grab technique can be found in many of the standard method texts.1-3/ HCBD in water samples is concentrated by extraction with appropriate organic solvents. Ether and n-hexane extraction of HCBD in water has been reported by Lebedeva et al.4/ and Timofeeva and Shvartsman,5/ respectively. In some cases, HCBD is concentrated by passing the water sample through a column filled with an appropriate trapping medium. The compound was then extracted by treating with acetone and hexane. This kind of concentration technique, with other trapping media such as activated charcoal, and polystyrene copolymer, Amberlite XAD-2 and XAD-4, has been successfully applied in trapping other chlorinated pesticides.6-11/

Sampling of HCBD in air is generally carried out by trapping the compounds either in an appropriate organic solvent or in an appropriate organic resin. The sampling of HCBD by bubbling air through two impingers filled with organic solvents such as benzene, ethanol, petroleum ether, cyclohexane, and hexane has been reported.12-14/ Columns of wood-charcoal cigarette-filter in series as well as silica gel have been used to trap HCBD in air.4,13,14/ The HCBD is recovered by appropriate solvent extraction. Organic resins such as Chromosorb A and Chromosorb 101 have been used to trap HCBD and other chlorinated pesticides.15,16/ A nylon-chiffon cloth (0.25 or 0.5 m^2) impregnated with ethanediol and held vertically in a wooden frame and exposed to the atmosphere for 5 days has been reported to trap organochlorine as well as organophosphorus compounds.17/ Another organic resin, Tenax®-GC, has been reported to be an efficient trapping medium for aromatic hydrocarbon and organochlorine.18/

WATER SAMPLING AND RECOVERY STUDIES

As a result of the literature search, it was decided that for water sampling, the "grab" method would be employed as the primary method. If the concentration of HCBD was suspected to be low, an Amberlite XAD-4 column would be used to concentrate the substance. Both sampling techniques were evaluated prior to actual field sampling.

Hexane Extraction

Table C-1 show the results of recovery studies for n-hexane extraction of HCBD from water samples fortified with 1 to 30 μg/liter. Each of the water samples (1 liter) was extracted with three 10-ml aliquots n-hexane, made up to the mark of a 100-ml volumetric flask, and analyzed for HCBD by gas chromatography. The average recovery was over 80% for HCBD.

Table C-1. RECOVERY STUDIES OF HCBD BY n-HEXANE EXTRACTION

Sample	Amount in water (μg)	Amount found in n-hexane (μg)	% recovery
1	1	0.8	80
2	2	1.5	75
3	3	2.3	77
4	5	4.5	90
5	10	8.6	86
6	20	17.2	86
7	30	23	77
Blank	None	None	

Elution from Amberlite XAD-4

Water samples fortified with 1 to 30 μg/liter of HCBD were passed through Amberlite XAD-4 columns containing 7 g of the trapping material. Recovery of the two substances was accomplished by eluting, first with a small amount of acetone, followed by a larger volume of n-hexane. Table C-2 shows that the average recovery of HCBD is close to 60% (first five runs).* The relatively lower recovery observed for HCBD, i.e., close to 60%, indicated that a significant amount of HCBD was lost, probably due to volatilization. Volatilization losses were eliminated when a closed system was used. The recovery of HCBD from the solvent eluent of the Amberlite XAD-4 improved to 93% (Run 6) with the closed system.

Table C-2. RECOVERY OF HCBD FROM WATER BY CONCENTRATION ON XAD-4

Run (μg/ℓ)	Flow rate (ml/min)	Recovery from n-hexane elution (%)	Recovery from Soxhlet extraction	Recovery from water extraction
1 (30)	2	57	0	Trace
2 (30)	2	56	0	0
3 (1)	2	60	Trace	Trace
4 (5)	2	45	Trace	0
5 (30)	8	67	0	Trace
6 (30)[a]	10	93	Trace	0

a/ Closed elution system.

---

* When actual environmental samples were analyzed, the amount of Amberlite XAD-4 used in sampling (250 g) was so high that recovery by elution was very time-consuming. Elution was replaced by overnight Soxhlet extraction.

Evidence of significant volatilization loss of HCBD was further demonstrated by the results of the following experiment. Five 250 ml water samples fortified with 5 μg/liter HCBD were placed in five 250 ml separatory funnels. Three of the funnels were left uncapped overnight while the remaining two funnels were capped. Each water sample was then extracted and analyzed for HCBD. The results, shown in Table C-3, indicate that appreciable amounts of HCBD were lost due to volatilization from the open system.

Table C-3. LOSS OF HCBD DUE TO VAPORIZATION

Run	Separatory funnel	HCBD recovery (%)
1	Capped	72
2	Capped	74
3	Uncapped	10
4	Uncapped	11
5	Uncapped	17

AIR SAMPLING AND RECOVERY STUDIES

Chromosorb 101 and Tenax®-GC were tested for their trapping efficiency utilizing a device shown in Figure C-1. This device was initially designed to check the recovery of HCBD from water by vaporization at reduced pressure. The results also indicate efficiency for collecting HCBD from water-saturated air.

One liter of water, fortified with 1 to 30 μg/liter of HCBD, was placed in a one-neck 24/40 flask with a thermometer pit so that the water temperature was monitored. The vapor was drawn through a Tenax®-GC or Chromosorb 101 (approximately 1 g) column with a vacuum pump or water aspirator. The vapor flow rate through the trapping column was monitored with a calibrated flowmeter and was maintained at 3 liters/min. After passing a known volume of vapor through the column, the trapping material was first extracted with n-hexane in an ultrasonic bath, then by overnight Soxhlet extraction. The remaining water was also extracted with n-hexane. All the extracts were analyzed for HCBD by electron capture gas chromatography.

Table C-4 shows the results of a set of seven experiments. Runs 1 and 2 were designed to compare the efficiency of Chromosorb 101 and Tenax®-GC, while Runs 3 through 7 were repeated experiments to evaluate the efficiency of Tenax®-GC at various HCBD concentrations. In general, under these experimental conditions, the trapping and recovery of HCBD with Tenax®-GC is more effective than with Chromosorb 101.

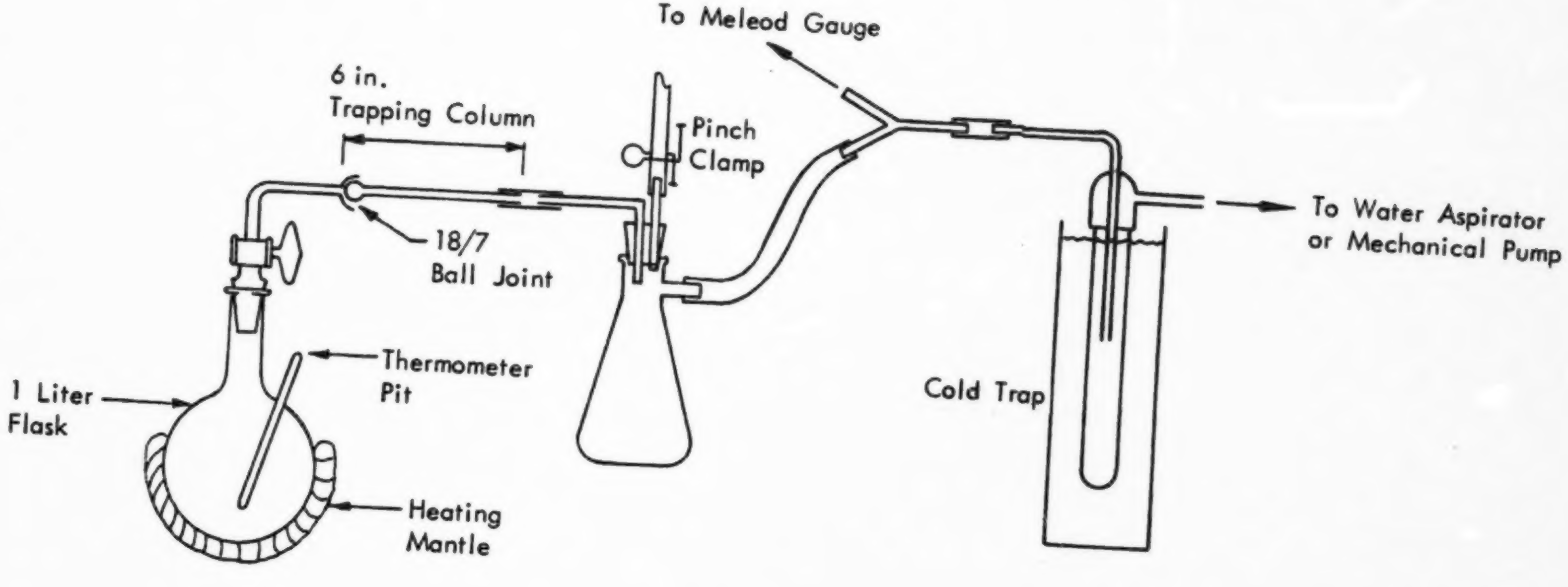

To Meleod Gauge
6 in.
Trapping Column
Pinch
Clamp
18/7
Ball Joint
To Water Aspirator
or Mechanical Pump
1 Liter
Flask
Thermometer
Pit
Cold Trap
Heating
Mantle

Figure C-1. Apparatus for recovery of HCBD from water by vaporization at reduced pressure.

Table C-4. RECOVERY OF HCBD FROM WATER-SATURATED AIR

Run	Column	Amount of HCBD in sample	% Recovery from ultrasonic extraction	% Recovery from Soxhlet extraction	% Recovery from extraction of water
1	Chromosorb	30	5	None detected	12
2	Tenax®-GC	30	50	Trace	4
3	Tenax®-GC	1	70	0	Trace
4	Tenax®-GC	1	88	Trace	Trace
5	Tenax®-GC	5	78	Trace	1
6	Tenax®-GC	30	79	1	2
7	Tenax®-GC	30	85	1	1

SEDIMENT SAMPLING AND RECOVERY STUDIES

Sediment was taken from two Kansas City area creeks. The samples were collected from the top 1 to 2 in. of sediment. The samples were fortified with HCBD and recoveries were determined using standard procedures (for sediments) described in the Manual of Analytical Methods prepared by the Pesticides and Toxic Substances Effects Laboratory of the National Environmental Research Center, USEPA. A Florisil™ column was used for sample cleanup and 6% ethyl ether in petroleum ether was used for the elution of HCBD.

Two different procedures were used to prepare fortified sediment samples. In the first method, HCBD was added to known amounts of sediment prior to evaporation of moisture from the sediment; in the second method HCBD was added after the moisture in the sediment had evaporated almost to dryness. Results of the recovery studies of these sediment samples (Runs 1 through 4) are shown in Table C-5.

Because of the low recovery of HCBD in Runs 1 through 4, direct Soxhlet extraction (1:1 acetone/hexane) of the fortified sediment samples was tested. Runs 6 and 7 in Table C-5 show the results of Soxhlet extraction. The recovery of HCBD improved significantly. Therefore, direct Soxhlet extraction of the sediment was chosen as the standard method. However, if interferences from other impurities were present, Florisil™ cleanup would be used. The amount of HCBD determined in the sample analysis was reported on dry weight basis.

Table C-5. RECOVERY OF HCBD FROM SEDIMENTS

Run	Sample weight (g)	Amount HCBD added (μg)	% Recovery (HCBD)
1[a]	50	5	22
2[a]	50	5	24
3[b]	50	5	40
4[b]	50	5	35
5	50	Control	ND
6[c]	50	5	83
7[c]	50	5	79

---

a/ HCBD added before moisture in sample was almost evaporated to dryness.

b/ HCBD added after moisture in sample was almost evaporated to dryness.

c/ Direct Soxhlet extract of sample.

REFERENCES FOR APPENDIX C

1. *Standard Methods: Water and Wastewater*, 13th Edition, APHA (1971).

2. *ASTM Standards, "Water--Atmospheric Analysis*," Part 23 (1972).

3. Cox, G. V., *Amer. Lab.*, **6**(7):36 (1974).

4. Lebedeva, T. A., et al., *Metody Anal. Pestits*, pp. 57-59 (1970) in Russian.

5. Timofeeva, O. A., and G. A. Shvartsman, *Novye Metody Tekhnol. Konstr. Vinodel. Proizvod*, pp. 117-119 (1972) in Russian.

6. Hyndshaw, A. Y., *J.A.W.W.A.*, **64**:309 (1972).

7. Buelow, R. W., J. K. Carswell, and J. M. Symons, *J.A.W.W.A.*, **65**:57 (1973).

8. Ibid., **65**:195 (1973).

9. Burnham, A. K., G. V. Calder, J. S. Fritz, G. A. Junk, H. J. Svec, and R. Willis, *Anal. Chem.*, **44**:139 (1972).

10. Kennedy, D. C., *Environ. Sci. Technol.*, **7**(2):138 (1973).

11. Burnham, A, K., G. V. Calder, J. S. Fritz, G. A. Junk, H. J. Svec, and R. Vick, *J.A.W.W.A.*, **65**:722 (1973).

12. Asriev, E. A., *Metody Anal. Pestits*, pp. 8-11 (1970) in Russian.

13. Dranovskaya, L. M., and A. G. Gul'ko, *Ostsillogr. Peremen. Polyarografiya*, pp. 163-167 (1971).

14. Gul'ko, A. G., L. M. Dranovskaya, and V. F. Chernokan, *Aktual. Vop. Gig. Epidemiol.*, pp. 71-73 (1972).

15. Mann, J. B., H. F. Enos, J. Gonzalez, and J. F. Thompson, *Environ. Sci.* Technol., **8**(6):584 (1974).

16. Aue, W. A., and P. M. Teli, *J. Chromatogr.*, **62**(1):15-27 (1971).

17. Tessari, J. D., and D. L. Spencer, *J. Assoc. Offic. Anal. Chem.*, **54**(6):1376-1382 (1971).

18. Enagonio, D. P., W. E. May, and S. P. Cram, Paper presented in the 25th Pittsburgh Conference on Analytical Chemistry and Applied Spectroscopy (1974).